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# MATERIALS FOR CRYOGENIC WIND TUNNEL TESTING

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National Bureau of Standards  
Boulder, Colorado 80303

Prepared for:

National Aeronautics and Space Administration  
Systems Engineering Division  
Langley Research Center  
Hampton, Virginia 23365

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## FOREWORD

The information in this report derives from literature surveys as well as inputs from NBS scientists with experience in cryogenics. The data presented are believed to be typical of materials in common forms and conditions. There is always some uncertainty in material properties, but errors can be greatly reduced by making sure that the materials used in design are equivalent in composition and condition to the materials for which handbook data are available. In this report some materials are classified as unacceptable for general cryogenic applications, based on conventional criteria. However, the "unacceptable" materials may be satisfactory for specific applications or exceptional circumstances, if suitability can be demonstrated by qualification tests.

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The author is indebted to L. L. Sparks, F. R. Fickett, J. G. Hust (Thermophysical Properties Division, NBS) and M. B. Kasen (Fracture and Deformation Division, NBS) for contributing information from their unpublished review manuscripts in the areas of instrumentation, electrical properties, thermal properties, and structural composites, respectively.

## TRADE NAME DISCLAIMER

The use of tradenames of specific products in this paper is essential to facilitate an understanding of the materials described. The use of tradenames for this purpose in no way implies endorsement or exclusive recommendation by NBS. Alternative materials and commercial suppliers are listed in buyers' guides such as the Materials Selector which appears annually in the Journal of Materials Engineering.



# TABLE OF CONTENTS

	Page
1. INTRODUCTION	1
1.1. The National Transonic Facility	1
1.2. Purpose of This Materials Study	2
2. MODEL DESIGN CONSIDERATIONS	3
2.1. Conventional Structural Design Approach	3
2.2. Recommended NTF Structural Design Approach	3
2.3. Fracture Mechanics	3
2.4. Alloy Selection for Fracture-Resistant Design	5
3. CRYOGENIC PROPERTY TRENDS	7
3.1. Tensile Properties	7
3.2. Fatigue Properties	7
3.3. Charpy Impact and Fracture Toughness Parameters	12
3.4. Elastic Properties	12
3.5. Electrical Resistivity and Conductivity	14
3.5.1. Metal Conductors and Alloys	14
3.5.2. Dielectrics (Insulators)	19
3.6. Thermal Expansion and Contraction	19
3.7. Thermal Conductivity	23
3.8. Martensitic Phase Transformation	26
3.8.1. Selection of Stable Stainless Steels	28
3.8.2. Selection of Metastable Stainless Steels	28
4. METALS AND ALLOYS FOR STRUCTURAL APPLICATIONS	30
4.1. Carbon and Alloy Steels	30
4.2. Nickel Alloy Steels	30
4.2.1. Austenitic Fe-Ni Alloys	32
4.2.2. Ferritic Fe-Ni Steels	33
4.2.3. 18Ni Maraging Steels	34
4.2.4. Other Ferrous Alloys	35
4.3. Stainless Steels	35
4.4. Aluminum and Its Alloys	41
4.5. Copper and Its Alloys	46
4.6. Nickel and Its Alloys	48
4.7. Titanium and Its Alloys	50
4.8. Cobalt and Magnesium	53
5. METALS FOR BRAZING AND SOLDERING	54
5.1. Fillers and Fluxes	54
5.2. Brazing	54
5.3. Soldering	56
5.4. Joints	59
6. METALS FOR PLATING AND PROTECTION	60

7.	NONMETALS	Page 61
7.1.	Wood	61
7.2.	Glass	62
7.3.	Ceramics	68
7.4.	Polymers	69
7.5.	Filled Polymers	72
8.	FOAMS, FILMS, AND FIBERS	75
8.1.	Foams	75
8.2.	Films	75
8.3.	Fibers	75
9.	STRUCTURAL COMPOSITE LAMINATES	79
9.1.	Industrial Polymer Laminates	79
9.2.	Low Pressure Polymer Laminates	79
9.3.	Metal Matrix Laminates	84
9.4.	Selective Reinforcement	84
10.	ADHESIVES, SEALANTS, AND RELATED MATERIALS	85
10.1.	Adhesives	85
10.2.	Sealants	86
10.3.	Potting Compounds	86
10.4.	Contact Agents	86
10.4.1.	Varnish	86
10.4.2.	Grease	87
11.	SEALS	88
11.1.	Nondemountable Seals	88
11.2.	Demountable Seals	88
11.2.1.	Design Considerations	88
11.2.2.	Gasket and O-Ring Seals	88
11.2.3.	Temperature-Assisted Seals	89
11.2.4.	Spring and Pressure-Assisted Seals	89
11.3.	Seal Materials	89
11.3.1.	Elastomers	90
11.3.2.	Other Polymers	90
11.3.3.	Metals	90
12.	FRICTION, WEAR, AND LUBRICATION	92
12.1.	Design Considerations	92
12.2.	The Necessity of Dry Lubrication	92
12.3.	Friction Coefficients	93
12.3.1.	Unalloyed Metals	93
12.3.2.	Alloys	93
12.3.3.	Lamellar Solids	95
12.3.4.	Polymers	95
12.3.5.	Filled Polymers	97
12.3.6.	Polymer Films and Coatings	97
12.3.7.	Ceramics, Cermets, and Glass	97

13.	INSTRUMENTATION	Page
13.1.	Temperature Sensors	99
13.1.1.	Thermocouples	99
13.1.2.	Resistance Thermometers	99
13.1.3.	Bondable Temperature Gages	102
13.1.4.	Thermistors	102
13.1.5.	Filled Systems	102
13.1.6.	Combined Thermometers	102
13.2.	Strain Gage Sensors	102
13.2.1.	Gage Selection	103
13.2.2.	Gage Factors	103
13.2.3.	Apparent Strain	103
13.2.4.	Self-Temperature Compensation	103
13.3.	Pressure Sensors	104
13.4.	Sensor-to-Specimen Contact	104
13.4.1.	Thermal Anchoring	104
13.4.2.	Thermal or Electrical Contact Agents	104
13.5.	Signal Maintenance	105
13.5.1.	Wire Insulation	105
13.5.2.	Wire Connections	105
13.5.3.	Feed-Throughs	105
13.5.4.	Shielding and Grounding	105
13.5.5.	Switching	107
14.	SOURCES OF CRYOGENIC DATA	108
14.1.	Technical Journals	108
14.2.	Monographs	108
14.3.	Conference Proceedings	108
14.4.	Computerized Data Banks	108
14.5.	Abstracting Services	109
15.	REFERENCES	110



# MATERIALS FOR CRYOGENIC WIND TUNNEL TESTING

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A study was conducted to guide the evaluation and selection of materials and techniques to be used in construction of model aircraft for cryogenic wind tunnel testing. In this report, the mechanical, thermal, and electrical property behavior of materials at temperatures as low as 77 K is briefly reviewed. Metals, structural alloys, non-metals, composites, joining methods, coatings, sealants, adhesives, contact agents, lubricants, transducers, and instrumentation for cryogenic applications are discussed. Acceptable structural materials, conductors, and insulators are discussed for service at temperatures in the range 367 to 77 K. Numerous references to handbooks and other cryogenic data sources are cited as a guide to additional information.

Key words: Cryogenic properties; instrumentation; low temperature materials; materials properties; metals; non-metallic materials; wind tunnels.

## 1. INTRODUCTION

A wind tunnel is an aerodynamic flow loop for testing model aircraft under simulated flight conditions to improve the performance and efficiency of full-scale aircraft. One relevant design parameter is the chord Reynolds number which characterizes the fluid flow past a structure by relating the velocity, density, and viscosity of the fluid to the dimensions of the body being tested. Unfortunately, conventional wind tunnels using air at room temperature are not able to simulate the very high Reynolds numbers experienced by modern aircraft operating in the transonic flight velocity range (Mach 0.8 to 1.2). This limitation of conventional wind tunnels has severely hampered aerodynamic engineering for the transonic range.

A unique solution to the problem was demonstrated in 1972 when NASA constructed a prototype (1/3-m square) cryogenic wind tunnel, proving that the necessary high Reynolds numbers could be obtained using nitrogen gas at temperatures as low as 80 K. In fact, a tenfold increase in Reynolds numbers is possible, owing primarily to the higher density and viscosity of cryogenic nitrogen compared to room temperature air. Cryogenic wind tunnels also enable independent control of Reynolds number, Mach number, temperature, and pressure. Thus, the effects of these variables can be separated and studied for the first time [1,2].

### 1.1. The National Transonic Facility

To take advantage of the benefits of cryogenic testing, NASA is currently constructing a National Transonic Facility (NTF). The NTF incorporates a 60-m-long, fan-driven, closed-circuit, high-speed cryogenic wind tunnel capable of generating Mach numbers from 0.2 to 1.2. The 2.5-m square test section accommodates models built on a 1:10 scale, or up to 1.5-m wingspan. The projected test pressures range from 100 to 900 kPa, while temperatures from 77 to 367 K may be achieved.



Cooling in this facility will be achieved by evaporating liquid nitrogen into the tunnel circuit at flow rates of up to 450 kg/s. Low temperature tests will require 3 h, with a gradual cool-down to 80 K taking 2 h. After reaching thermal equilibrium at the test temperature, the velocity of the circulating gas will be gradually increased. Severe impact loading, such as encountered in some conventional blow-down tunnels, will be avoided, but the static loads imposed on models and structural supports will be up to five times higher compared with conventional wind tunnels.

## 1.2. Purpose of This Materials Study

Owing to differential thermal contraction and the embrittling effects of low temperatures, combined with extremely high stress, it is certain that many traditional model construction materials and fabrication techniques are unacceptable for the newly developed NTF applications. To date, model design has been based entirely on experience gained at room or elevated temperatures. Reliable design practices must now be developed for the cryogenic region.

This study was undertaken to provide guidance in the evaluation, selection, and use of materials for NTF model building. The text is addressed to design engineers who may be totally unfamiliar with cryogenic technology. Contained here is an evaluation of potentially useful materials and techniques, representative data for specific materials of interest, notes on acceptable cryogenic practice, and recommendations intended to support materials selection and the development of design criteria for NTF models and structural supports.



## 2. MODEL DESIGN CONSIDERATIONS

Wind tunnel models incorporate a broad range of materials and fabrication methods. Several models may be needed for each proposed design or test temperature range. The models often have moving or interchangeable parts, and most are instrumented for pressure, temperature, or strain measurements. As listed in Table 2.1, metals and alloys are the most widely used construction materials. Wood, glass, ceramics, polymers, and composites have miscellaneous applications.

### 2.1. Conventional Structural Design Approach

Wind tunnel models are traditionally designed by experience, intuition, and stress calculations using safety factors of 3 on yield stress and 4 on ultimate tensile strength. Fatigue resistance is evaluated using smooth or notched bar S versus N curves. This approach assumes stresses are approximately uniformly distributed over a given cross section, so that safety factors are sufficient to mask differences between the assumed and actual stress distributions. The approach is best suited to small, ductile components.

### 2.2. Recommended NTF Structural Design Approach

The conventional design approach outlined above is adequate for many NTF structures, particularly secondary structures. However, safety factors of 3 or 4 are restrictive and impractical for critical primary components (e.g., model stings, balances, and fuselage struts). In these structures, the design allowable stresses are projected to range up to 700 MPa, as opposed to 193 MPa for conventional models. The higher stresses coupled with safety factors of 3 or 4 require a performance exceeding the limits of currently available structural alloys. It is, therefore, recommended that reduced safety factors be permitted for primary components and that fracture mechanics be employed to ensure structural safety. Safety factors as low as 1.5 are used in some cryogenic aerospace components that are subject to fracture mechanics analysis [3]. Fracture mechanics has not previously been used in model design, but is recommended for NTF model design since:

- 1) For many alloys, low temperatures tend to increase the probability of brittle fracture,
- 2) High design stresses tend to increase crack initiation and growth rates in structures subject to low-cycle fatigue,
- 3) Primary structures require high-strength alloys that are flaw sensitive, and
- 4) Model fractures would be costly in terms of damage to the NTF itself.

### 2.3. Fracture Mechanics

The application of fracture mechanics to cryogenic structures is reviewed by McHenry [4]. The goal of fracture mechanics is accurate component life prediction. Typically, a small crack is assumed to exist in a structure at a critical location. The material properties of interest then are the yield stress ( $\sigma_y$ ), the plane strain fracture toughness ( $K_{IC}$ ), and the fatigue crack growth rate behavior ( $da/dN$  versus  $\Delta K$ ).

The stress intensity factor,  $K$ , is proportional to the magnitude of elastic stresses at the crack tip. In service,  $K$  increases as the load increases, or as the crack grows larger during fatigue. Failure occurs when the critical value,  $K_{IC}$ , is reached. Using handbook equations for specific component geometries, the maximum flaw size that a cracked structure can tolerate can be calculated based on the material's  $K_{IC}$  value. Fatigue crack growth rates then enable a prediction of the total number of stress cycles (lifetime) required to propagate an initial or assumed crack to critical size.

The initial assumption that a flaw exists in the structure is made for the sake of conservative life prediction. The assumption is particularly appropriate for high strength alloys, welded structures, or structures subject to low-cycle fatigue.

Table 2.1. Materials for wind tunnel model construction.

Applications	Materials used in conventional wind tunnels at 295 K	Materials recommended for cryogenic wind tunnels at 77 K
Primary and secondary structures	Carbon steel Alloy steel Stainless steel Superalloys Aluminum alloys	Nickel alloy steels Austenitic stainless steels Nickel superalloys Titanium alloys Aluminum alloys
Secondary structures	Composites Copper alloys Polyamides	Composites Copper alloys
Electrical conductors or insulators	Precious metals Copper alloys Ceramics Glass Polyamides Fluoroplastics	Precious metals Copper alloys Ceramics Glass Fluoroplastics
Thermal conductors or insulators	Copper alloys Ceramics Glass Elastomers Polyamides Reinforced plastics Fluoroplastics	Copper alloys Ceramics Glass Reinforced plastics Fluoroplastics
Lubricants	Oils, grease Precious metals Dry films	Dry films
Seals	Fluoroplastics Elastomers	Fluoroplastics Metals
Adhesives, fillers	Epoxies	Epoxies, modified epoxies
Plating	Nickel, chromium Precious metals	Nickel Precious metals

## 2.4 Alloy Selection for Fracture-Resistant Design

It may be assumed that the fatigue crack growth rates of alloys from a particular base metal system are nearly equivalent [5]. Initial alloy selection is then based on consideration of yield stress and fracture toughness. As shown in Fig. 2.1,  $\sigma_y$  and  $K_{Ic}$  are inversely related, with high strength alloys exhibiting lower toughness. Therefore a proper balance between these parameters must be obtained for a particular application.

A general discussion of how  $\sigma_y$  and  $K_{Ic}$  influence the design approach is given by Crooker and Lange [5]. Wells, Kossovsky, Logsdon, and Daniel [6] provide one example of a parametric analysis illustrating alloy selection and life prediction. These references provide a starting point for engineers seeking familiarity with fracture mechanics and alloy selection. A figure of merit in alloy selection is the crack size factor,  $(K_{Ic}/\sigma_y)^2$ . This factor is proportional to the critical flaw size for a given material, and it accounts in a single term for the effects of both strength and toughness on flaw tolerance [7]. The best material for a given application is the one having the highest crack size factor, assuming other requirements (e.g., cost, availability, etc.) are satisfied. The absolute value of critical flaw size must be calculated for each application, because it depends on the applied stress level and component geometry, as well as on material properties.

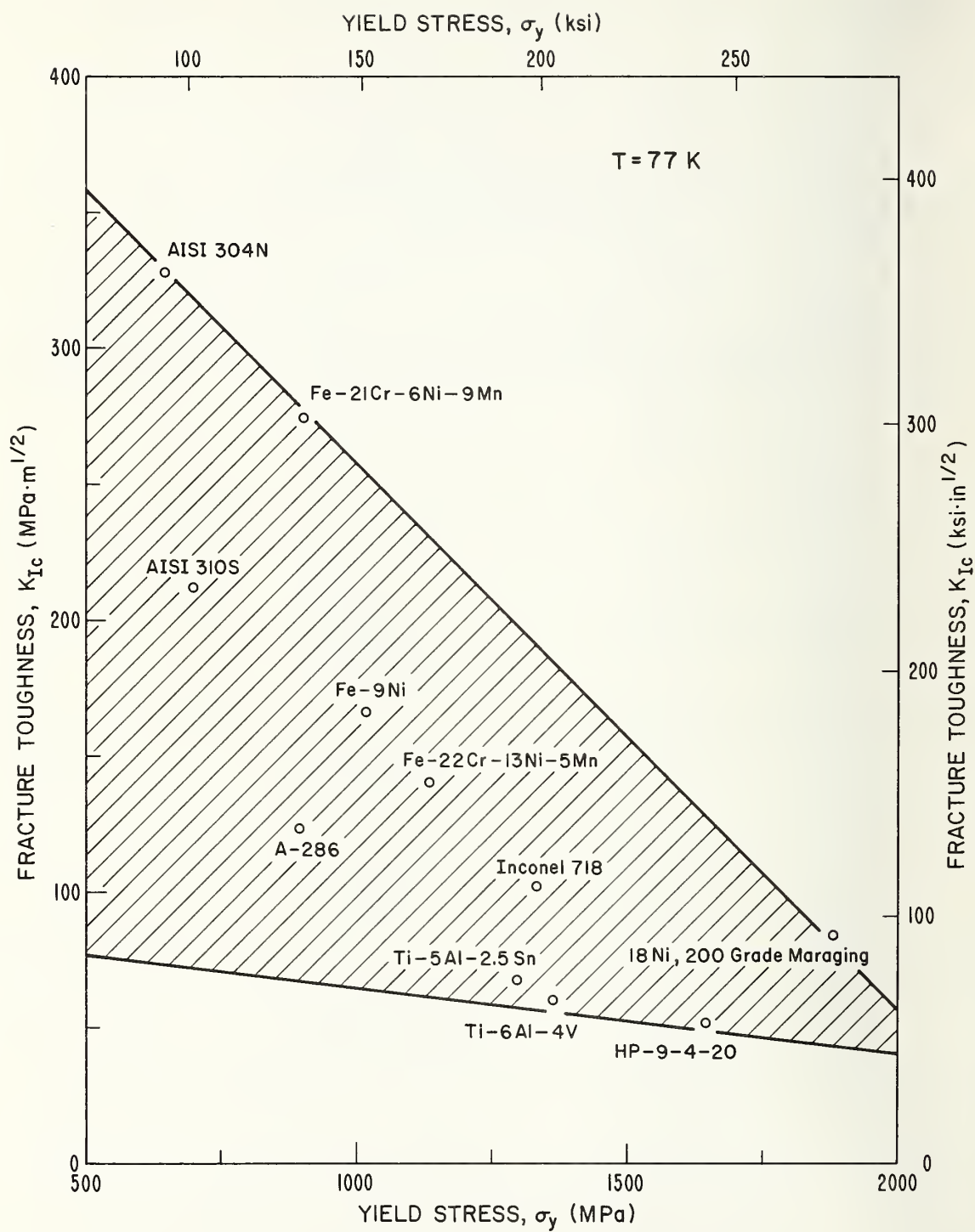


Figure 2.1. Toughness-versus-strength trend for structural metals at 77 K.



### 3. CRYOGENIC PROPERTY TRENDS

This chapter describes the expected temperature dependence of mechanical, thermal, and electrical properties of interest to model design. Selected design parameters, data trends, data sources, measurement uncertainties, and data estimating techniques are discussed. Cryogenic data handbooks [8-17] are recommended as primary data sources, whereas Wigley's text [18] is recommended as a basic introduction to cryogenic property behavior. In this chapter, the aluminum alloy system is used to exemplify the regular cryogenic behavior of face-centered cubic metals. Specific materials recommendations are given in subsequent chapters.

#### 3.1. Tensile Properties

ASTM Method E8-69 [19] describes standard procedures for measuring the yield and ultimate stresses and ductility of metals and alloys in the unnotched condition. The 0.2% yield stress is a quantitative design parameter, while reduction of area and elongation values are qualitative guides to brittle fracture resistance. The uncertainty in yield stress values owing to material variability and measurement errors is typically less than  $\pm 10\%$ .

Tensile property data are compiled in several handbooks [8-13,17]. Materials generally show increased strengths at low temperatures (Fig. 3.1), but ductility may increase or decrease. Cryogenic effects on metals and alloys are summarized by Wigley [18], Conrad [20], and Wessel [21]. Face-centered cubic (fcc) metals and alloys show desirable behavior at low temperatures, whereas body-centered cubic (bcc) or hexagonal close packed (hcp) metals are embrittled. The classical trends for an fcc metal are as shown in Fig. 3.2 for aluminum: reduction of area is high, elongation increases, there is a modest increase of yield stress and a greater increase of ultimate strength. As a result, austenitic stainless steels and alloys of copper, aluminum, and nickel are widely used for cryogenic service.

In contrast to fcc metals, bcc and hcp metals show a stronger temperature dependence of yield stress, in conjunction with low temperature embrittlement. Embrittlement usually takes the form of a ductile-to-brittle transition, which is marked by an abrupt loss of ductility, impact energy, toughness, and fatigue resistance occurring over a narrow temperature interval. Most commercially available ferritic or martensitic steels, bcc refractory metals, bcc titanium alloys, tetragonal tin or tin-based solders, and most polymers exhibit low temperature transitions, which render them useless for cryogenic service. But a few ferritic and martensitic materials are useful at 77 K, as discussed in section 4.2.2. and 4.2.3.

#### 3.2. Fatigue Properties

Fatigue is a process of cyclic plastic deformation resulting in crack initiation, crack propagation, and fracture on the final stress cycle. Both crack initiation and propagation occur in conventional fatigue tests which report stress-versus-cycles-to-failure (S-N) data for smooth or notched specimens. S-N results for some cryogenic metals and alloys are reported in the literature [22-29].

Under stress-controlled conditions, the fatigue resistance of metals and alloys usually shows improvement at 77 K, compared with 295 K. Exceptions to this behavior are bcc alloys, which are embrittled at low temperatures, and metastable austenitic stainless steels, which exhibit martensitic transformation. Under strain-controlled fatigue conditions, a cross-over behavior is typical--e.g., cycle life at 77 K is usually higher than at 295 K for high-cycle fatigue, whereas the reverse is true for low-cycle conditions (Fig. 3.3).

In fracture mechanics, fatigue crack propagation is treated separately, as discussed by Clark [30]. Fatigue crack growth rates ( $da/dN$ ) are plotted versus the fracture mechanics parameter  $\Delta K$ , which is the difference between the maximum and minimum stress intensity factors produced by the peak loads of the fatigue cycle. Typical data for cryogenic alloys are shown in Fig. 3.4. In general, temperature effects on  $da/dN$  behavior correlate with crystal structure [31]:

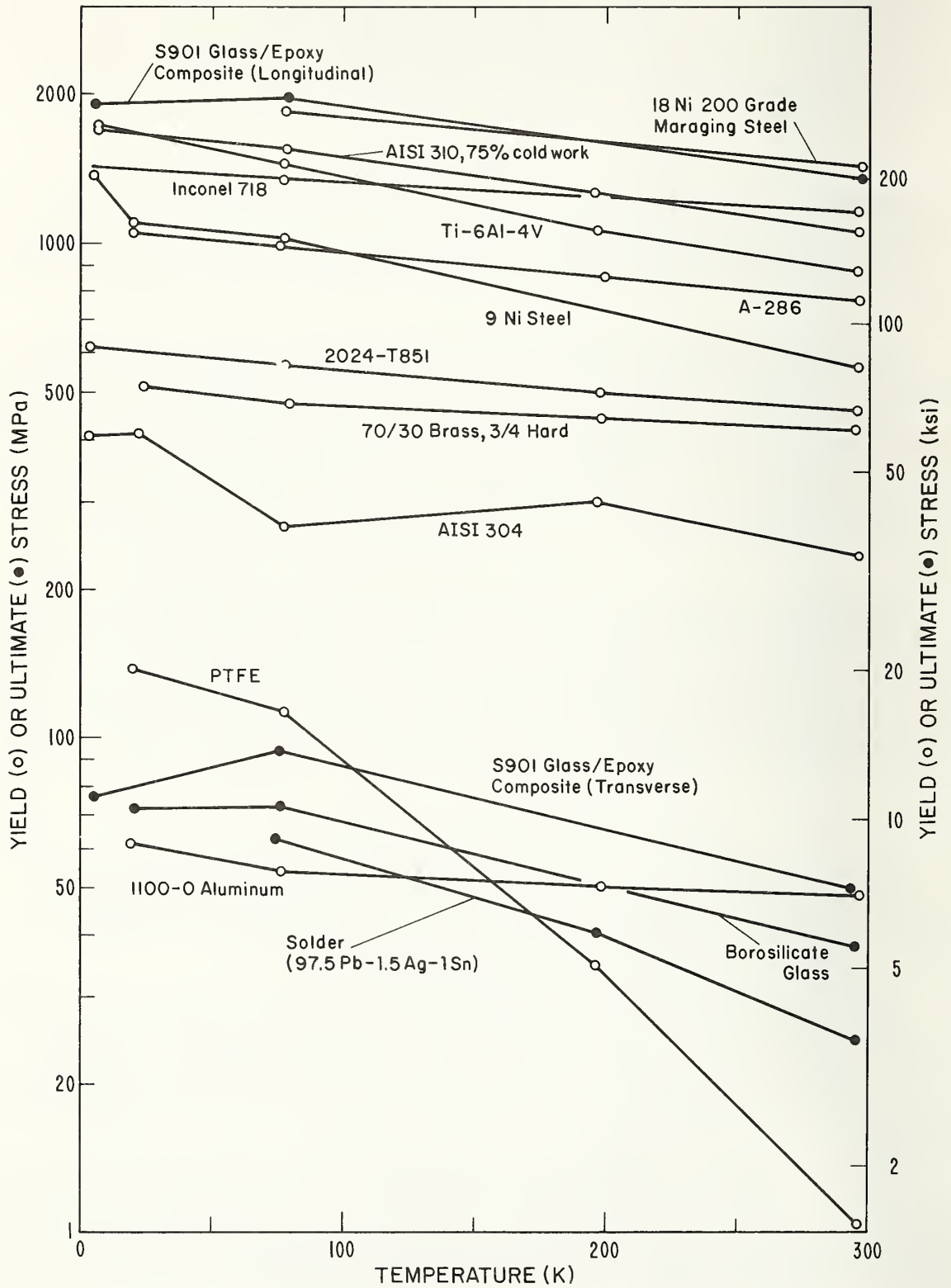


Figure 3.1. Cryogenic effects on strength.

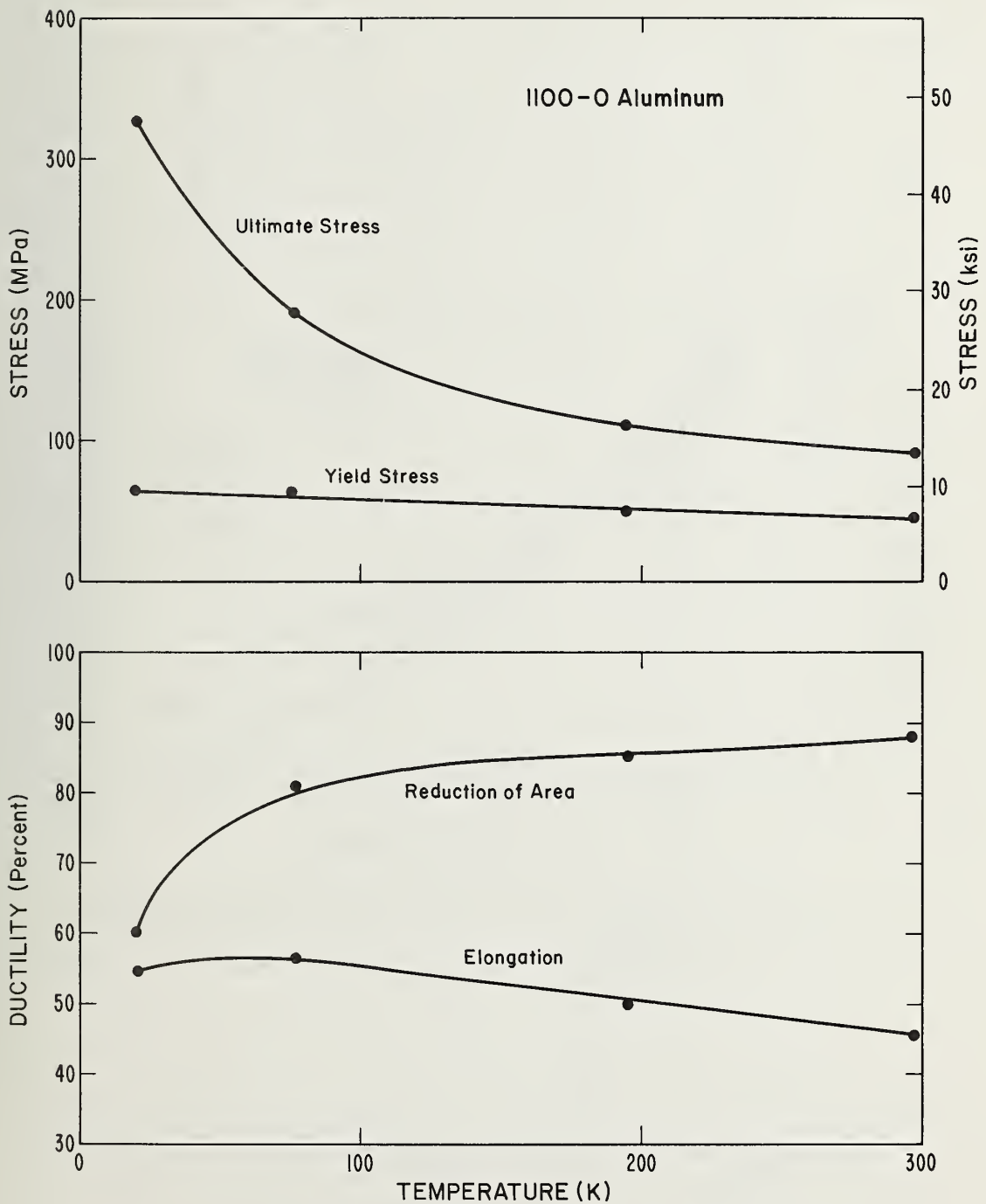


Figure 3.2. Cryogenic effects on the tensile properties of an aluminum (fcc) alloy.

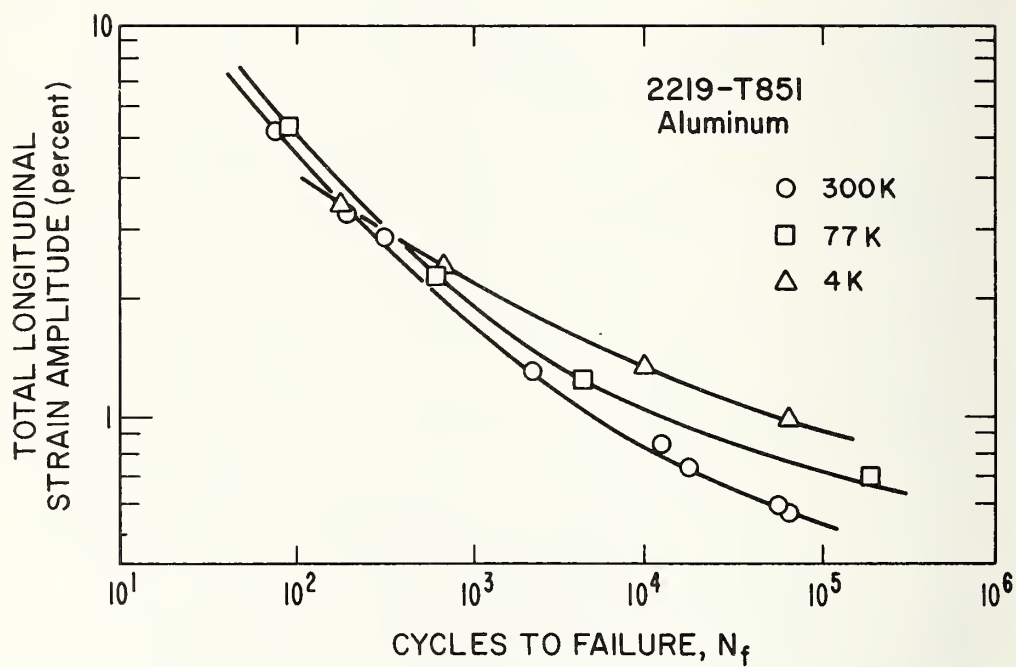


Figure 3.3. Cryogenic effects on the fatigue life of an aluminum (fcc) alloy.



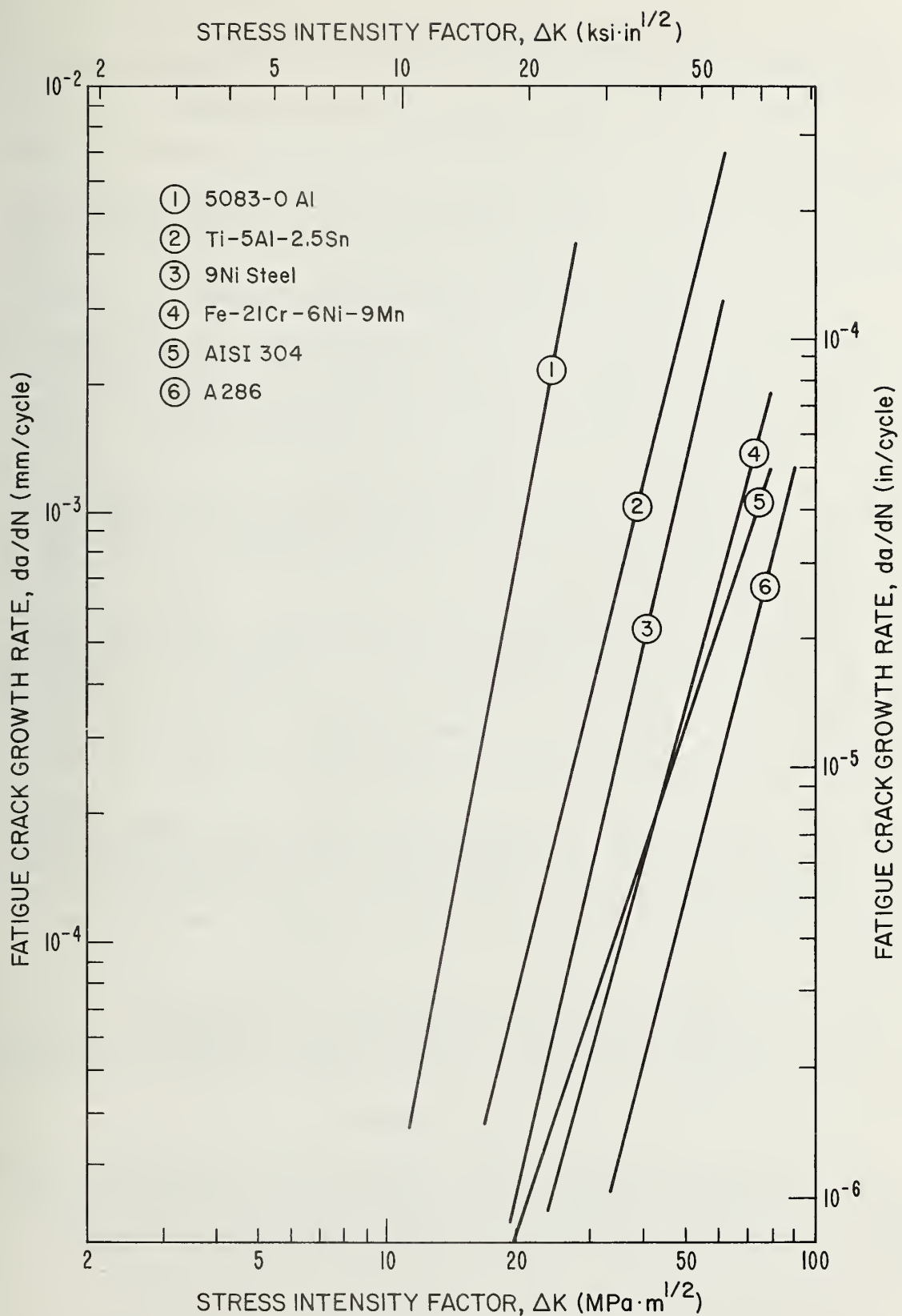


Figure 3.4. Fatigue crack growth rates for structural alloys at 77 K.

- 1) Fcc alloys show reduced rates at low temperature,
- 2) Hcp titanium alloys show no measurable temperature dependence,
- 3) Bcc alloys and ferritic steels show increased rates at low temperatures where cleavage begins to occur, and
- 4) Metastable austenitic stainless steels show unpredictable behavior.

Inconel superalloys and stable austenitic stainless steels exhibit the greatest resistance to fatigue crack growth at 77 K.

Scatter in fatigue data is high. In terms of the value of  $da/dN$  at a given  $\Delta K$  value, the interlaboratory variability may be 3:1, while the intralaboratory variability may be 2:1 [32].

### 3.3. Charpy Impact and Fracture Toughness Parameters

In the case of high strength alloys, uniaxial tensile ductility does not assure a ductile fracture mode if cracks are present. For example, fatigue-cracked Inconel 718 (fcc structure) fractures in a brittle manner at 295 and 76 K, even though 20% elongation and 20% reduction of area are observed in conventional tensile tests. This crack-sensitive behavior is typical of all high-strength alloys and is attributed to the fact that cracks introduce a nonuniaxial stress state locally.

Charpy V-notch (CVN) impact values and notched tensile tests are traditionally used for the purpose of ranking the toughness of structural materials. But the plane strain fracture toughness,  $K_{IC}$ , is a more sophisticated toughness parameter that can be used quantitatively to calculate the allowable stress levels for components in service, in addition to ranking the toughness of materials.

$K_{IC}$  is measured according to ASTM Method E 399-72 [33]. Valid results are achieved only if the specimen fails under linear-elastic conditions. The uncertainty in valid  $K_{IC}$  measurements is between 4 and 10% [34]. Estimates of  $K_{IC}$  based on the J-integral are less accurate, about  $\pm 15\%$ . Moreover,  $K_{IC}$  values are highly sensitive to composition, specimen orientation, and heat treatment. Therefore, designers must be sure that any handbook values of  $K_{IC}$  are representative of the alloy in service.

$K_{IC}$  data for some structural alloys of cryogenic interest appear in some handbooks [10-12], but no single data source is adequate. Most cryogenic data have only recently been generated, so the best data sources may be conference proceedings and journal articles. Quantitative predictions of  $K_{IC}$  are unreliable, but a qualitative prediction of temperature effects on  $K_{IC}$  can be based on alloy crystal structure. Representative trends are shown in Fig. 3.5. In general:

- 1) Fcc alloys retain relatively high toughness at all temperatures,
- 2) Hcp titanium alloys have relatively low toughness at all temperatures, and
- 3) Bcc alloys show abrupt transitions from high to low toughness at critical temperatures associated with the onset of cleavage. Charpy impact data usually show a temperature dependence similar to  $K_{IC}$ .

### 3.4. Elastic Properties

The most useful elastic constant is Young's modulus, which is routinely used to calculate the deflection and buckling resistance of components under elastic stress. Young's modulus for a material may be measured as the initial linear slope of the engineering stress-strain curve, but acoustic measurement methods provide greater accuracy. The engineering values are accurate to  $\pm 10\%$ , whereas acoustic measurements are accurate to  $\pm 2\%$ .

Values of  $E$  are more commonly quoted, but Poisson's ratio ( $\nu$ ), shear modulus ( $G$ ) and bulk modulus ( $B$ ) are also available in many publications cited. If only two of the elastic constants are known, the others can be calculated from the following equations, assuming isotropic behavior:

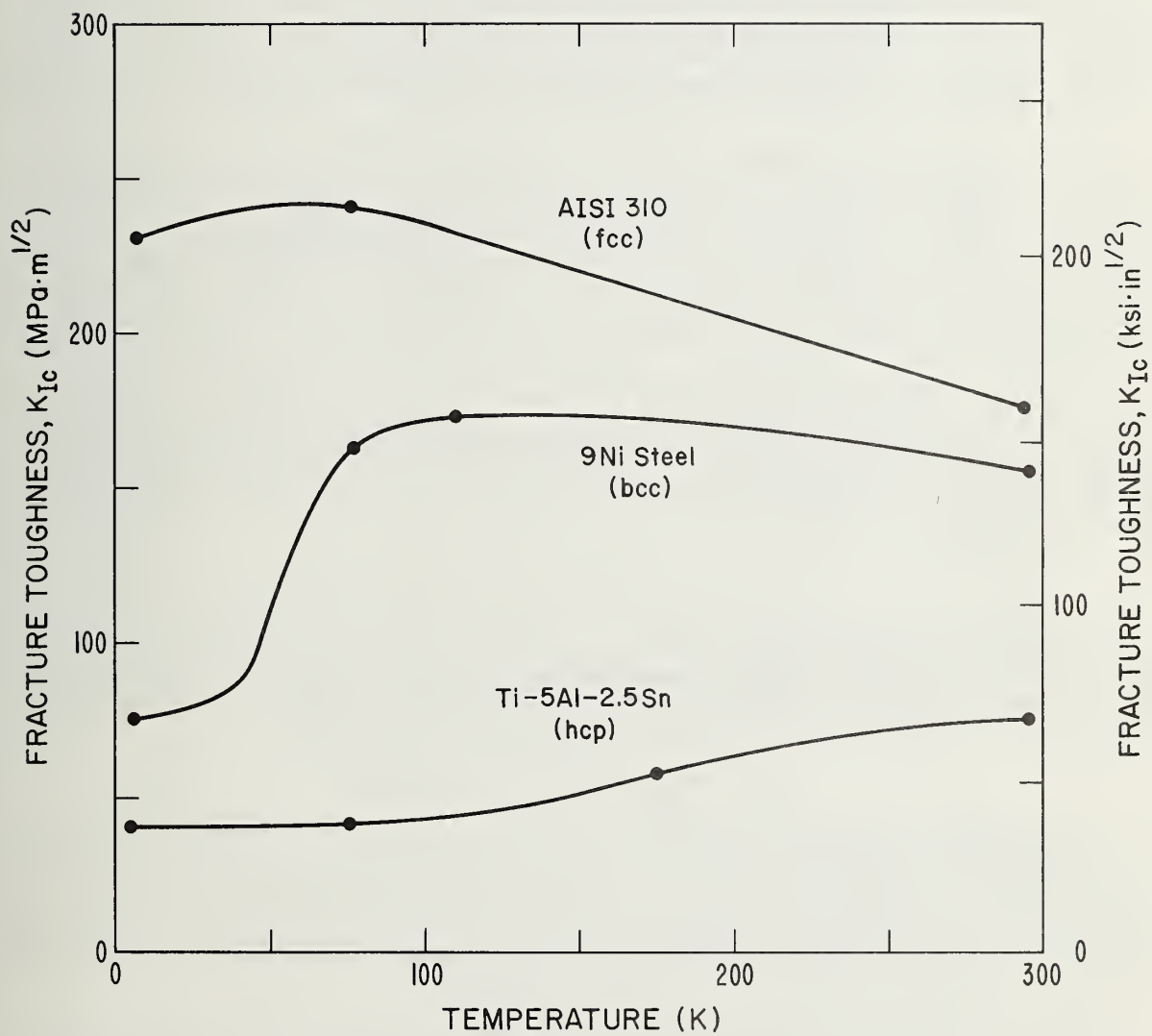


Figure 3.5. Cryogenic effects on fracture toughness of structural alloys.

$$G = \frac{E}{2(1 + \nu)}$$

$$B = \frac{E}{3(1 - 2\nu)}$$

$$E = \frac{3G}{1 + G/3B}$$

The elastic properties of metals and alloys are weakly dependent on cold work, heat treatment, and alloy phase or magnetic transformations. For example, aged maraging steels show  $E$ ,  $G$ ,  $\nu$ , and  $B$ , increases of 9, 10, 5, and 0.1%, respectively, as compared with the solution annealed condition. Material composition is a more important factor. In addition, porosity in wood or ceramics and fiber orientation in composite laminates may significantly influence the elastic properties.

Fig. 3.6 compares the Young's moduli for several materials as a function of temperature. The highest values are observed for some commercial ceramics, and the lowest, for polymers. Composites, metals, and glasses typically are intermediate. As temperature is decreased from 300 to 20 K, a ten-fold increase of  $E$  occurs for some polymers, whereas ceramics, metals, and glasses show no more than a several percent increase. Most materials exhibit a smooth and continuous modulus increase at cryogenic temperatures, but exceptions and anomalies are noted for unstable austenitic stainless steels, Invar, and glass. Anomalies occur in some austenitic stainless steels, owing to magnetic transitions [35].

In Fig. 3.7, the relationships between  $E$ ,  $G$ ,  $\nu$  and  $B$  are illustrated for a well-behaved metal, 2219-T8 aluminum [36].  $E$ ,  $G$ , and  $B$  increase at low temperatures, while  $\nu$  decreases. For fcc metals,  $G$  and  $E$  are expected to be proportional at a ratio of about 0.375. For metals and alloys exhibiting regular behavior, the nearly linear behavior between 100 and 295 K extends to higher temperatures, so that data can be extrapolated to 400 K for engineering purposes.

### 3.5. Electrical Resistivity and Conductivity

In addition to applications in electrical design, electrical resistivity ( $\rho$ ) and conductivity data are used to characterize the purity of metals and to predict values of other transport properties which are more difficult to measure, such as thermal conductivity. As shown in Table 3.1 there is an enormous resistivity difference amounting to a factor of  $10^{25}$  between metals (conductors) and non-metals (insulators).

#### 3.5.1. Metal Conductors and Alloys

Good electrical conductors are metals of commercial purity or better. The best electrical conductors at 295 K rank as follows: silver, copper, gold, aluminum. The electrical conductivities of metals improve at low temperatures, but not at identical rates. Thus, copper and aluminum rank highest at 76 K, and these two metals are the major competitors for wiring and electrical applications. Silver, platinum, palladium, and gold serve as contact terminals, but they are too expensive for broader use. Nickel is a fair conductor, finding some applications in electrical instruments.

For pure metals, the decrease in resistivity between 295 and 76 K is rather large, typically 80%. Structural alloys have much higher resistivities and show smaller changes, about 20 to 30%, for the same temperature range. Commercial resistance alloys are purposefully designed to be insensitive to temperature, showing changes as small as 0.5%. Resistance alloys find applications as precision resistors and strain gage elements, whereas some pure metals and dilute alloys are used for temperature sensing [37-43].

Representative data for pure aluminum and its alloys are shown in Fig. 3.8. Copper and its alloys behave similarly. High purity aluminum (99.99%) shows a continuous decrease

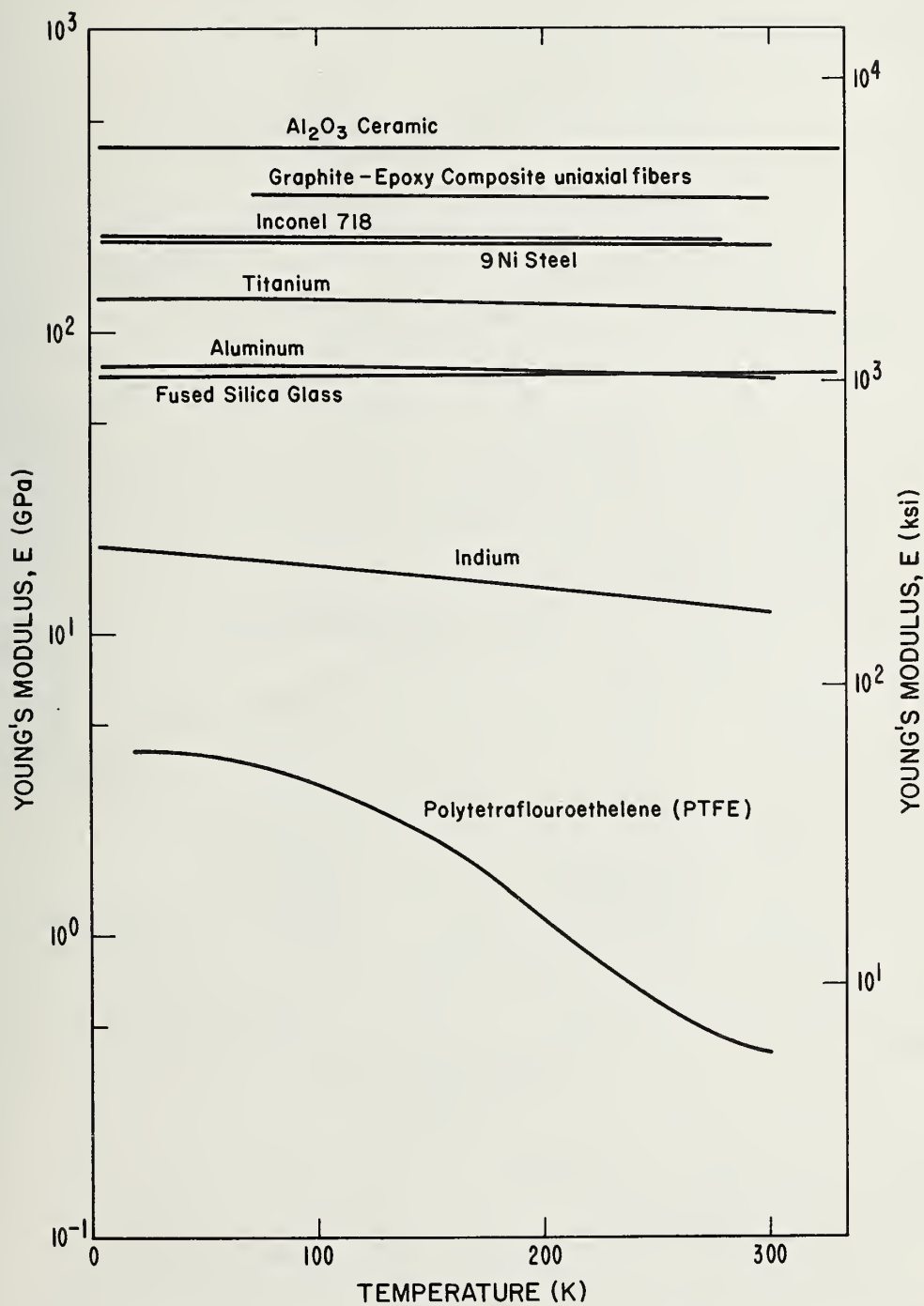


Figure 3.6. Cryogenic effects on Young's modulus of materials.

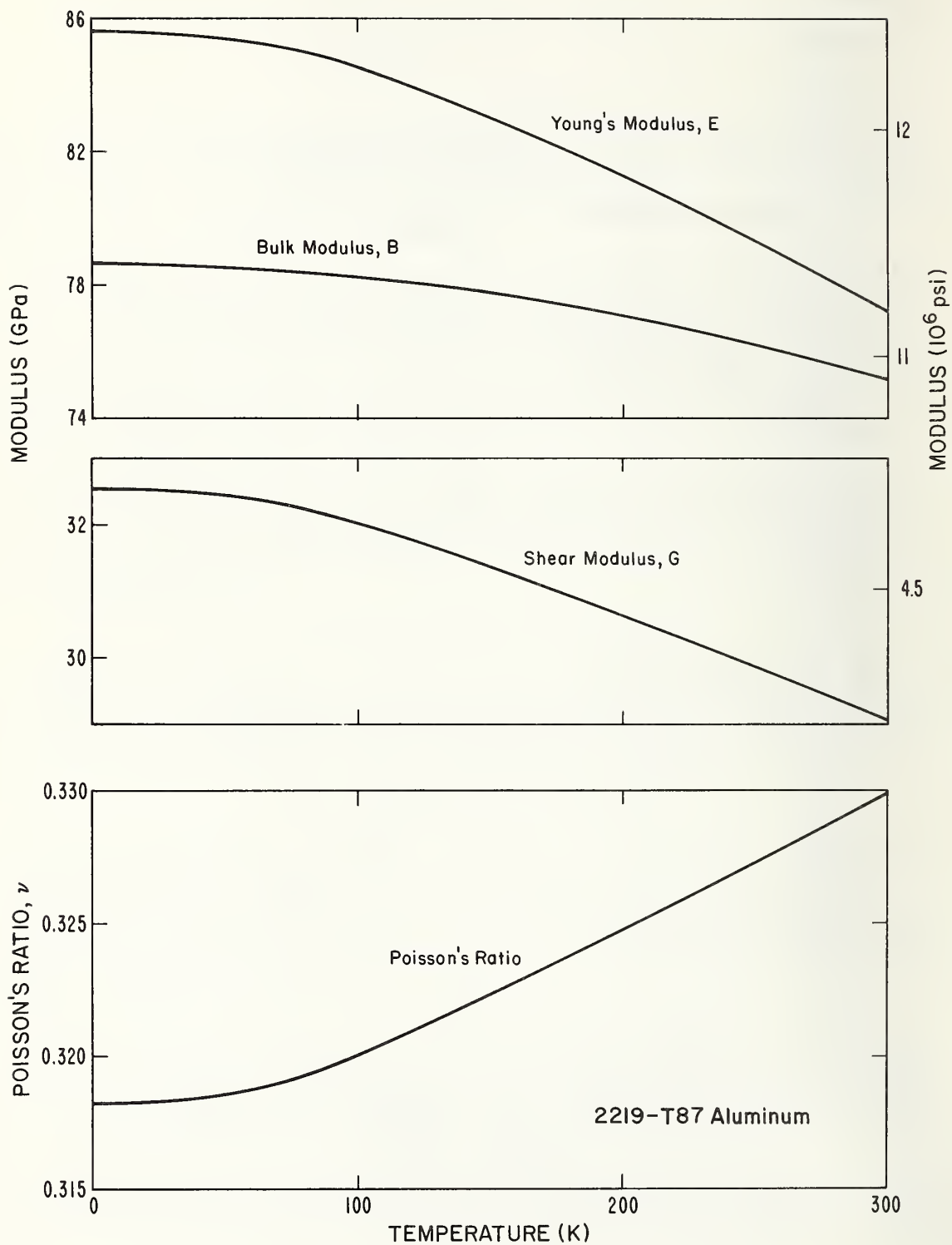


Figure 3.7. Cryogenic effects on the elastic constants of an aluminum (fcc) alloy.



Table 3.1. Resistivities of selected materials [37-41].

Materials	$\rho$ @ ~ 295 K ( $\mu\Omega\text{cm}$ )	$\rho$ @ 273 K ( $\mu\Omega\text{cm}$ )	$\rho$ @ 80 K ( $\mu\Omega\text{cm}$ )	$\rho$ @ 76 K ( $\mu\Omega\text{cm}$ )	% change
<u>Pure Metals</u>					
Silver	1.61	--	0.29	--	82
Copper	1.70	--	0.21	--	88
Gold	2.20	--	0.46	--	79
Aluminum	2.74	--	0.25	--	91
Nickel	7.00	--	0.55	--	92
Tin	11.0	--	2.10	--	81
Lead	21.0	--	4.97	--	76
<u>Commercial Metal Conductors</u>					
Copper, OFHC	--	1.559	--	0.20	87
Aluminum 1100-0	--	2.67	--	0.31	88
Nickel TD	--	6.73	--	0.74	89
<u>Structural Alloys</u>					
70:30 Brass	--	6.65	--	4.65	30
Beryllium Copper	--	9.21	--	7.24	21
304L Stainless Steel	--	70.4	--	51.4	27
Inconel X-750	--	123	--	118	4
Invar	--	79.5	--	54.4	31.5
Ti-6Al-4V, ELI	--	156	--	137	12
<u>Resistance Alloys</u>					
Evanohm (75Ni-20Cr, Al-Cu)	134	--	133	133	0.5
Karma (76Ni-20Cr, Fe, Al)	133	--		132	0.5
Constantan (57Cu-43Ni)	49	--		48	2
Manganin (84Cu-12Mn-4Ni)	48	--		45	6
<u>Insulators</u>					
Polyimide	$2 \times 10^{21}$	--	$>2 \times 10^{23}$	--	--
Polycarbonate	$3 \times 10^{21}$	--	$>2 \times 10^{23}$	--	--
Polyethylene terephthalate	$2 \times 10^{23}$	--	$>2 \times 10^{23}$	--	--
Polytetrafluoroethylene	$3 \times 10^{19}$	--	$4 \times 10^{18}$	--	--
Pyrex glass	--	$5 \times 10^{17}$	--	--	--

Note:  $1 \mu\Omega\text{cm} = 0.394 \mu\Omega\text{in.}$

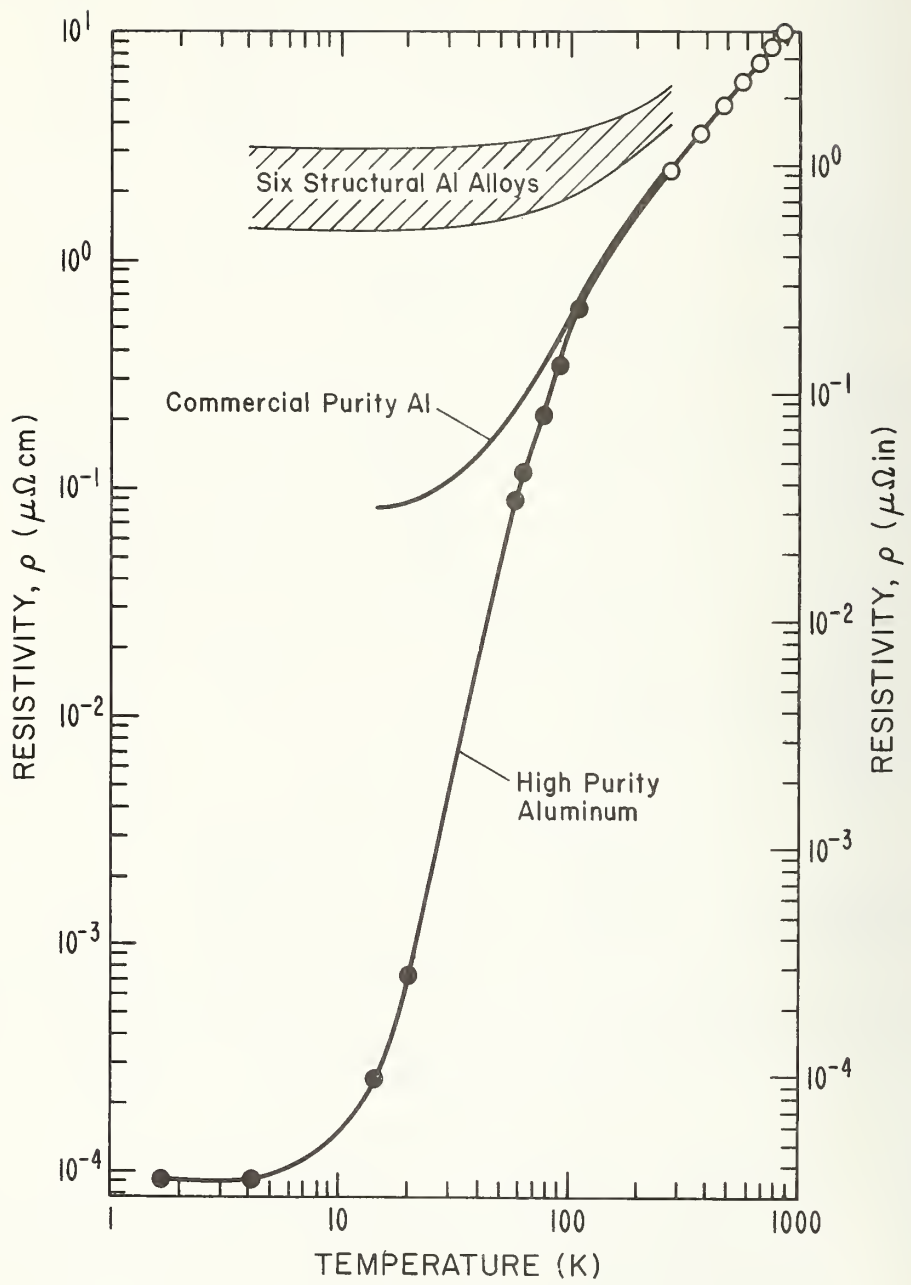


Figure 3.8. Electrical resistivity of aluminum alloys.



in resistivity as temperature is lowered, until a plateau is reached at about 10 K [38-40]. Adding even small amounts of impurities or alloy elements rapidly degrades the electrical conductivity such that the plateau value of resistance for commercially pure aluminum (99%) is three orders of magnitude greater than the value for high purity aluminum (99.99%). With further alloying, the plateau resistance becomes predominant. Thus, heavily alloyed structural materials exhibit high resistivities that are only mildly temperature dependent.

A comparison of alloys is shown in Fig. 3.9 [38]. Titanium alloys have the highest resistivities; next highest are the heavily alloyed superalloys, stainless steels, brasses, bronzes, cupronickels, aluminum alloys, and pure metals. Between 295 and 76 K, the temperature dependence of  $\rho$  for alloys is gradual and continuous, assuming it is not interrupted by phase transformations, such as occur in some metastable austenitic stainless steels. The trends reported by Clark, Childs, and Wallace [38] should facilitate prediction or extrapolation of the resistivities for untested alloys.

### 3.5.2. Dielectrics (Insulators)

The electrical resistivities of polymers and ceramics are so large that measurement is practically impossible because the sensitivity of common current measuring devices is exceeded [41, 42]. Thus, resistivity values for dielectrics at low temperatures are not widely available. But resistivity data for dielectrics are not needed in engineering design. NTF model testing entails small signal processing as in temperature and strain monitoring. Therefore, it is sufficient to note that the common electrical insulators remain good insulators at all temperatures.

### 3.6. Thermal Expansion and Contraction

The thermal expansion and contraction of materials must be accounted for in any multicomponent structure operating over a broad temperature range. The length change of a material during heating or cooling depends on its initial length, the temperature change involved, and its thermal expansion coefficient. Thermal expansion or contraction is described by either the total length change relative to room temperature ( $\Delta L/L$ ) or by the coefficient of linear expansion,  $\alpha$ , where

$$\alpha = \frac{1}{L} \frac{dL}{dT}$$

The most common measurement technique is the push rod dilatometer, which provides an uncertainty in  $\Delta L/L$  typically less than  $\pm 5\%$ .

The thermal contraction of metals at cryogenic temperatures is reported by Arp, Wilson, Winrich, and Sikora [44], Rhodes, Moeller, Hopkins, and Marx [45], and Clark [46]. Diller and Timmerhaus [47] review the behavior of engineering materials in general. In addition, several handbooks contain cryogenic data [15, 16]. Figure 3.10 compares the general trends of engineering materials. Polymers have high thermal contractions between 295 and 77 K, with  $\Delta L/L$  values about four times greater than typical metals and alloys and twenty times higher than glass. Low contraction materials include fused silica or high-silica glasses, graphites or glassy carbons, certain glass-ceramics, and Invar [16, 48].

As for Young's modulus, thermal expansion is a structure-insensitive property, strongly dependent on material composition and related to basic aspects of atomic bonding. Clark [46] observes that a rather large alloy addition of 10-20% may be needed to produce a 10% change in the thermal expansion for a particular base metal, and that heat treatment or processing do not have a large effect unless a phase change occurs, as in martensitic transformation or precipitation hardening. Aluminum provides a good example of regular behavior in a base metal system for which there are no known anomalies. As shown in Fig. 3.11, the thermal contractions between 293 and 77 K of unalloyed aluminum and a variety of commercial aluminum alloys agree to within  $\pm 7\%$ .

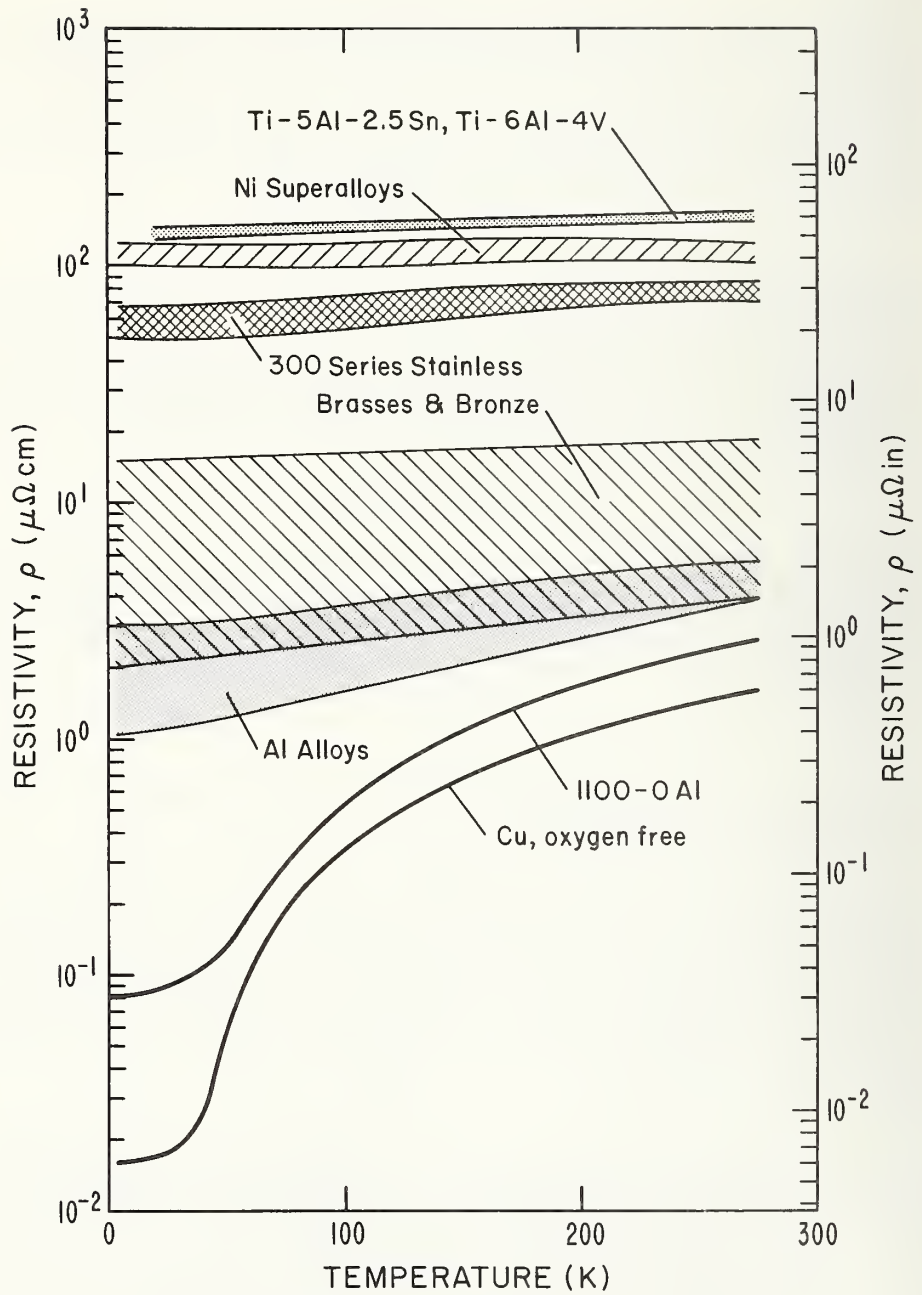


Figure 3.9. Cryogenic effects on electrical resistivity of metals.

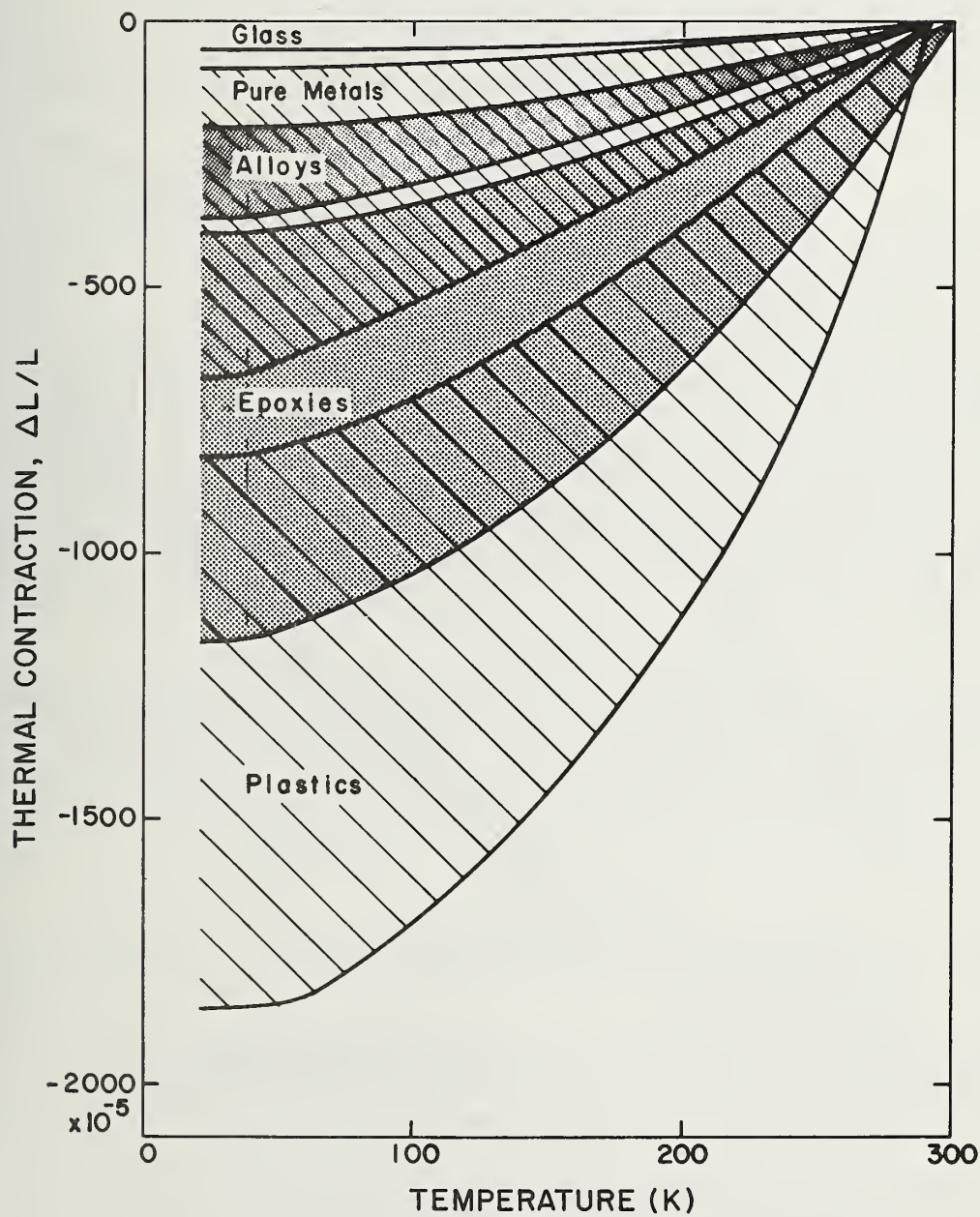


Figure 3.10. Cryogenic effects on thermal contraction.

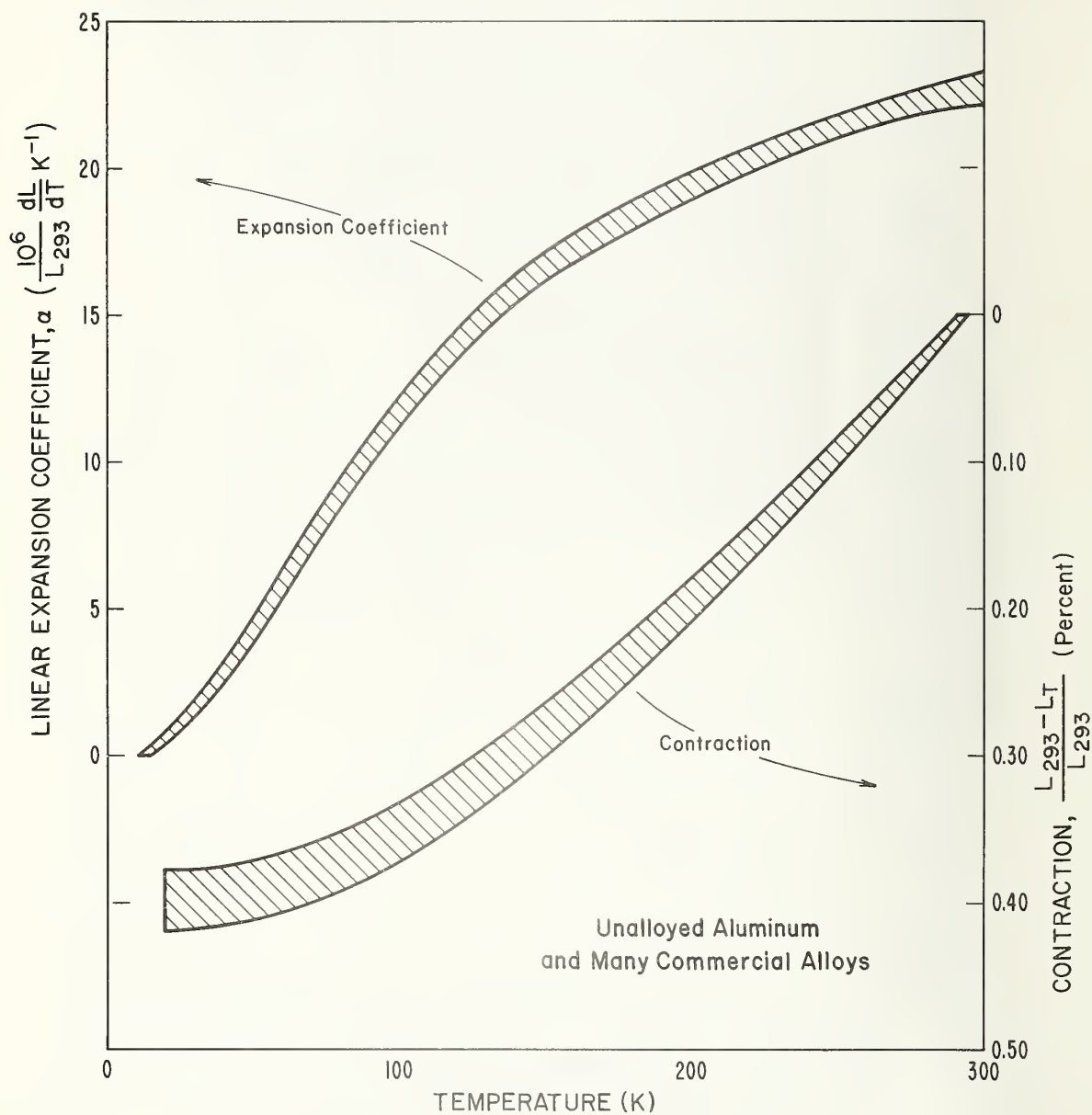


Figure 3.11. Cryogenic effects on the thermal contraction of aluminum alloys.



Although most solids contract on cooling, there are notable exceptions and anomalies:

- 1) Fused silica undergoes a reversal and begins to expand after cooling below 200 K.
- 2) Lithium-aluminum silicate is an inorganic material that expands on cooling below 295 K, and
- 3) Invar is an exceptional alloy that contracts only 0.05% between 295 and 77 K, whereas a 0.3% contraction is typical for steels.

The thermal expansion of untested materials may be predicted by several methods. First, there is a proportionality between  $\alpha$  and specific heat. This will yield an engineering estimate for  $\alpha$  at a specified temperature,  $T$ , assuming that the specific heat,  $C_v$ , is known at another reference temperature. Second, estimates can be based on data for similar materials as suggested by Fig. 3.11. A third approach for alloys is based on the ratio:

$$\frac{L_{77\text{ K}} - L_{295\text{ K}}}{\alpha_{295\text{ K}}}$$

For many alloys, this ratio falls in the range 170 to 190, so the value of  $\alpha$  at 295 K is a useful guide to the total contraction expected between room temperature and 77 K [46].

Unless thermal contraction in cryogenic apparatus is anticipated, a variety of undesirable effects will be encountered. Consequences range from simply a loss of machined tolerances to the introduction of residual stresses, buckling, or cracking. Fractures may develop when dissimilar materials are joined and cooled to cryogenic temperatures where incompatible strains are set up. Self-cracking is common in brittle bulk materials (glasses, polymers) subject to local, rapid cooling. To alleviate problems, thermally induced strain gradients must be reduced. The overlap of data bands in Fig. 3.10 suggests the possibility of choosing materials having matching thermal expansion characteristics. Other approaches include: (1) using plastics filled with low expansion glass, ceramic, or asbestos fibers or powders (2) using low expansion materials, (3) using thin section construction where possible, and (4) reducing thermal shock by gradual cool-down rates.

### 3.7. Thermal Conductivity

Hust [49] reviews thermal conductivity and thermal diffusivity considering theory, measurements, data trends, correlations and predictive techniques. Of particular significance to model designers engaged in heat transfer calculations is one-dimensional, longitudinal heat flow under steady state. For these conditions thermal conductivity is defined as the proportionality constant, between the heat flow rate ( $Q$ ) per unit area ( $A$ ) and the temperature gradient:

$$-\frac{Q}{A} = k \frac{\Delta T}{\Delta X}$$

The existing handbooks containing low temperature thermal conductivity data for materials are particularly valuable since they contain some data from unpublished sources and from corporate literature [10, 13-15].

Data trends for selected engineering materials are compared in Fig. 3.12. Reduced thermal conductivity at low temperatures is typical for most materials, but moderate increases between 300 and 76 K are observed for high purity metals. At temperatures of interest for NTF testing, the spread of thermal conductivity values for solids covers four orders of magnitude. Data for aluminum and its alloys are shown in Fig. 3.13.

Comparison of Figs. 3.12 and 3.9 reveals an inverse ranking of metals as regards electrical resistivity and thermal conductivity. This correlation does not carry over to nonmetals, however. Metals generally have much higher electrical conductivity than nonmetals, but there are certain ordered dielectrics (diamond, sapphire, beryllium oxide) that have thermal conductivities approaching or exceeding metals.

Thermal conductivity measurements are time consuming and expensive, and no single

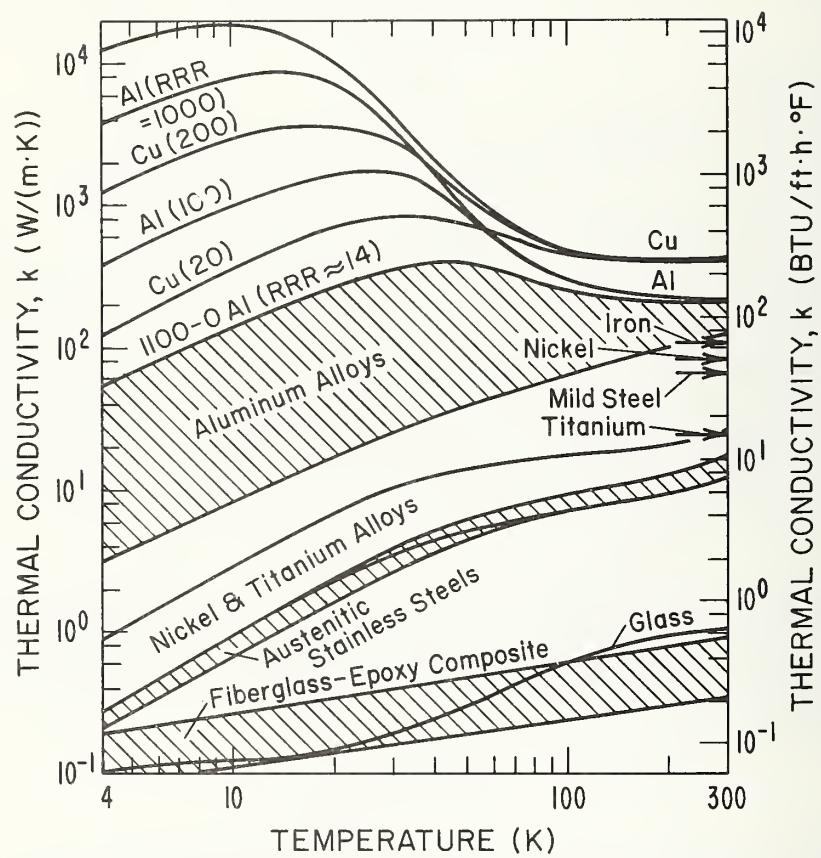


Figure 3.12. Cryogenic effects on thermal conductivity of materials.

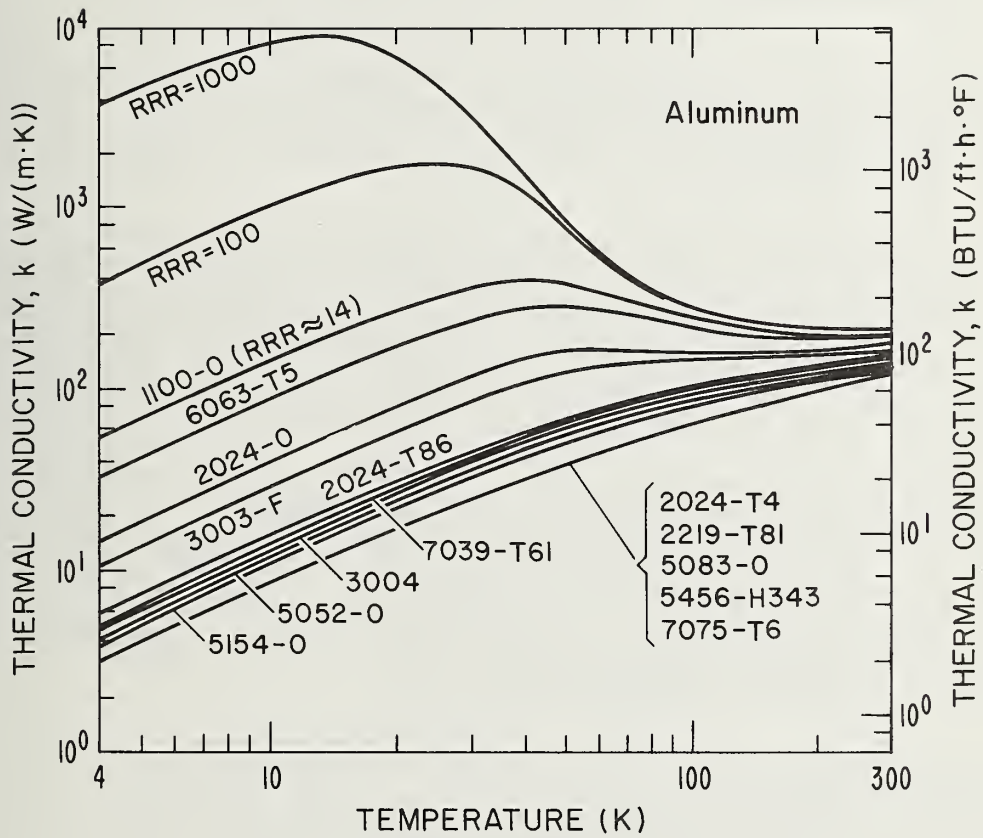


Figure 3.13. Cryogenic effects on the thermal conductivity of aluminum alloys.

measurement technique suffices for all material and temperature combinations. For precision apparatus and best technique, measurement uncertainties may be pushed as low as  $\pm 1\%$ , although  $\pm 10\%$  is more usual, and still higher uncertainties are recorded in specific cases. Material variability can also be great. For example, the thermal conductivity of high purity metals such as copper and aluminum may vary by orders of magnitude at temperatures below 76 K. Pure metals and structural alloys exhibit significantly reduced material variability at 76 K and above.

Predictions based on theory, empirical relationships, or existing data are of particular interest in this area because experimental results for many materials at cryogenic temperatures do not exist. Interpolated or extrapolated predictions based on existing data may be uncertain within  $\pm 5$  to  $\pm 50\%$  [49]. Predictions may be based on residual resistivity or Lorenz ratios in the case of metals and alloys [50], but the only reasonable prediction scheme for an untested nonmetal is comparison with a chemically similar material.

### 3.8. Martensitic Phase Transformation

Martensitic transformations occur in some alloys after cooling or deformation at low temperatures. This transformation consists of a sudden shearing of a group of atoms that alters the crystal structure locally, with no atomic diffusion. A variety of physical and mechanical property changes occurs in conjunction with martensitic transformation and these changes must be anticipated in design.

Reed and Breedis [51] review the subject of martensitic transformations. Most cryogenic metals and alloys (e.g., copper, aluminum, nickel) are crystallographically stable at all temperatures and exhibit no martensitic transformation. But austenitic stainless steels may transform. In austenitic stainless steels, the face-centered cubic phase (austenite) is metastable, being achieved by the alloy additions of Cr, Ni, Mn and other elements. But the equilibrium structure for iron-based alloys at room temperature and below is body-centered cubic. Therefore, transformation may occur during service, depending on alloy content, service temperature, stress level, and metallurgical condition.

In susceptible AISI 300 series stainless steels, the fcc phase ( $\gamma$ ) partially transforms to hcp ( $\epsilon$ ) and bcc ( $\alpha$ ) martensitic phases. These transformations may occur spontaneously on cooling, or they may be induced by application of stress. In the case of one AISI 304L alloy, about 2% martensite forms on cooling to 77 K, whereas 90% martensite forms with deformation to the point of fracture [52], as shown in Fig. 3.14. Spontaneous transformations have also been reported in AISI 303, 304, 321, and 347 stainless steels, and repeated thermal cycling may cause progressive transformation [53]. Among the consequences of transformation are volumetric expansion, magnetic transition, and anomalies in the temperature dependence of physical and mechanical properties.

Volumetric Expansion. Martensitic transformation in AISI 300 series stainless steels is accompanied by a volume expansion of about 3%. This expansion can be detrimental in some applications, causing loss of dimensional tolerances, loosening bolts, or creating residual stress. For example, transformation in a machined stainless steel airfoil could cause surface relief effects that would ruin the aerodynamic surface. Such problems are best overcome using stable alloys that do not transform.

Magnetic Transition. The bcc martensites formed in austenitic stainless steels are ferromagnetic whereas the hcp martensites and the original fcc phases are paramagnetic (non-magnetic). Ferromagnetic components may cause problems in magnetic fields. Where problems could develop, magnetic permeability should be considered. Stable stainless steels, such as AISI 310, have low magnetic permeability values in the range from 0.001 to 0.009, whereas AISI 304 grades containing several per cent bcc martensite have permeability values of about 1.0 at 77 K.

Mechanical Effects. Martensitic transformations in austenitic steels are capable of influencing the tensile, elastic, fatigue, and fracture properties. Deleterious effects on ductility and toughness at cryogenic temperatures might be expected, since the martensites formed have non-fcc crystal structures. In fact deleterious effects occur in association with transformation in the AISI 200 series stainless steels, which are not recommended for cryogenic service (section 4.3.).



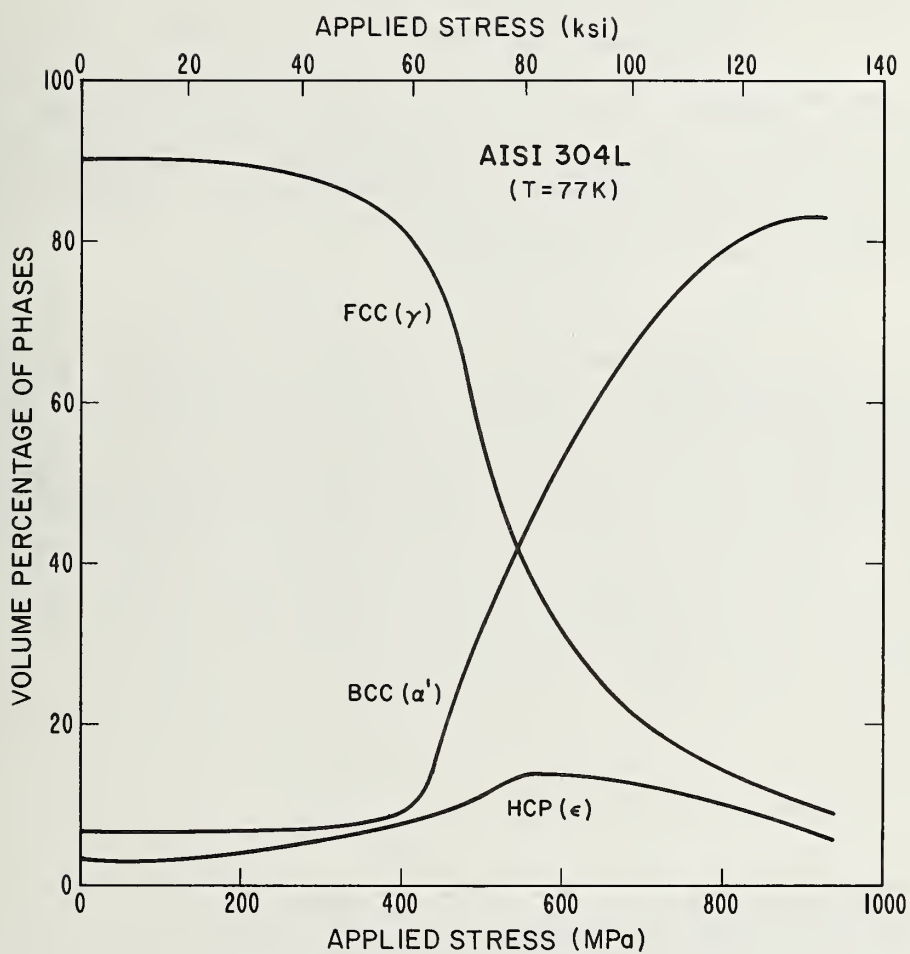


Figure 3.14. Stress effects on the martensitic phase transformation in an AISI 304L stainless steel.

In the case of AISI 300 series stainless steels, however, the effect of martensitic transformation on mechanical behavior is of little significance for routine base metal applications. Since the stability of the recommended AISI 300 series stainless steels is relatively high, little or no martensite is formed in the absence of extensive plastic deformation. With AISI 304, for example, different heats have varying tendencies to transform owing to compositional variations within the material specification, but excellent cryogenic tensile properties are expected with some scatter, as indicated by the data of Fig. 3.15. Other effects of martensitic transformations on mechanical and physical properties of austenitic stainless steels are noted in section 4.3.

### 3.8.1. Selection of Stable Stainless Steels

To avoid any possibility of martensitic transformation, one solution is to choose a fully stable alloy. AISI 310 (Fe-25Cr-20Ni) and A 286 (Fe-25Ni-14Cr) are examples of completely stable stainless steels which, owing primarily to high nickel content, can be deformed to fracture at 77 K or lower with no evidence of phase transformation. These alloys exhibit regular physical and mechanical property trends with temperature, which are similar to the usual trends for other stable fcc metals and alloys. In contrast, anomalous trends are expected for unstable stainless steels.

### 3.8.2. Selection of Metastable Stainless Steels

Another approach is to choose metastable alloys that are sufficiently stable for service conditions in a particular application. Williams, Williams, and Capellaro [54] and Larbalestier and King [55] compare the relative stabilities of AISI 300 stainless steels at temperatures between 4 and 373 K. Stability increases with total alloy content. Thus, AISI 316 is more stable than AISI 304, and high nitrogen grades such as AISI 304N are more stable than normal AISI 304 or low-carbon grade AISI 304L. The temperature at which martensite forms spontaneously on cooling ( $M_s$ ) can be predicted to within about 40 degrees K, based on composition [54, 56].

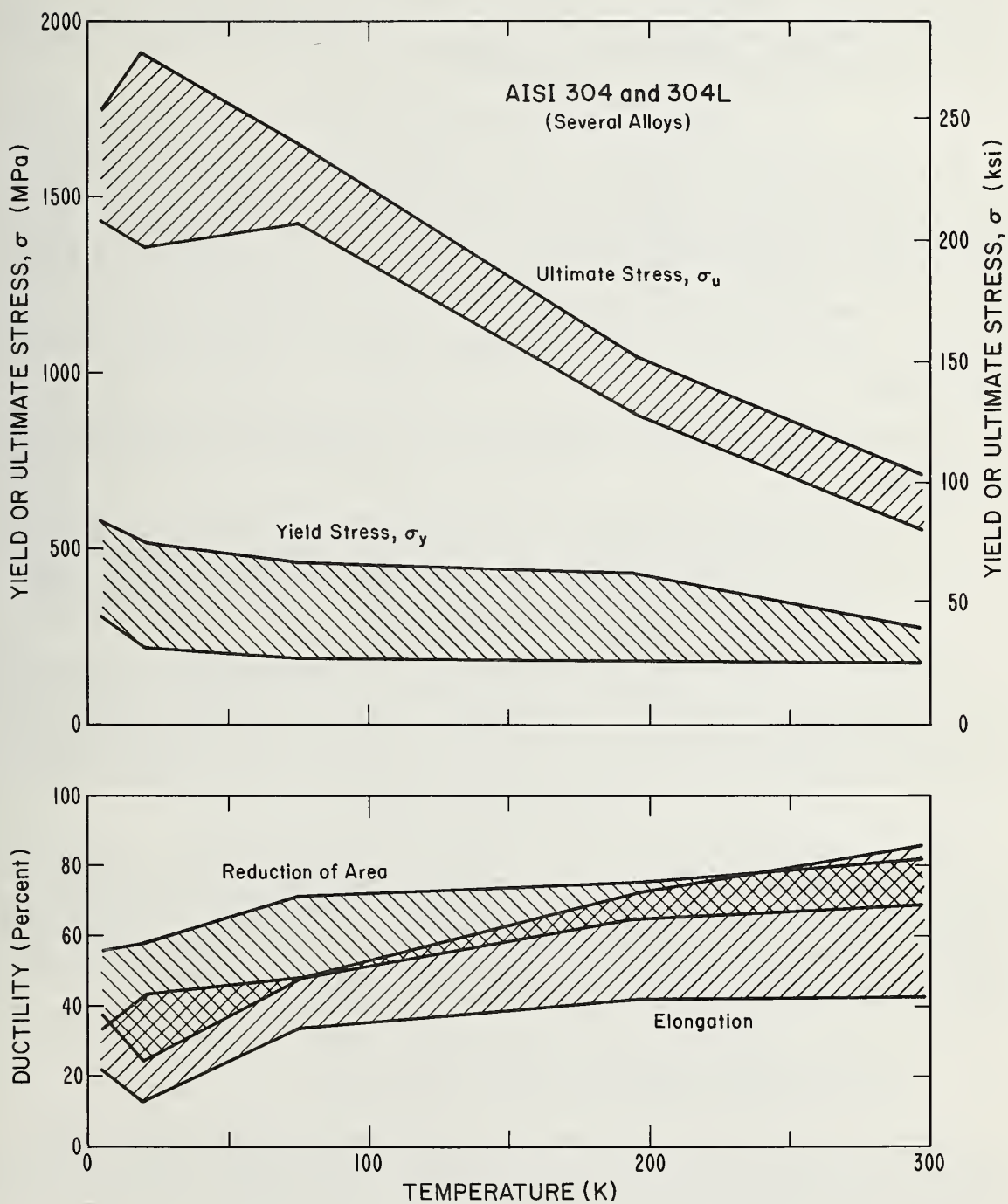


Figure 3.15. Scatter in tensile properties owing to phase transformations in AISI 304 stainless steels.

## 4. METALS AND ALLOYS FOR STRUCTURAL APPLICATIONS

Metals and structural alloys are the most widely used model materials. Castings are used when complex shapes are required, but wrought products are generally favored for lower cost and superior ductility. Suitability for service at cryogenic temperatures is largely dependent on composition and crystal structure. This chapter discusses metals at temperatures as low as 77 K, concluding that, as primary structural alloys, conventional carbon and alloy steels generally must be abandoned in favor of certain nickel alloy steels, austenitic stainless steels, and nonferrous fcc alloys.

### 4.1. Carbon and Alloy Steels

Owing to their non-fcc structures, carbon steels such as SAE 1020 (Fe-0.2C-1Mn) possess totally unacceptable cryogenic ductility and toughness. Such steels exhibit classical notched bar ductile-to-brittle fracture transitions at temperatures in the range of 250 to 375 K depending on material and test variables [57-59]. Above the transition temperature, ductile failure occurs by void coalescence; below the transition temperature, brittle failure occurs by cleavage with zero ductility. The Charpy impact energy for a mild carbon steel cleaving at 77 K is only 6.8 J, which corresponds to a  $K_{IC}$  value of only  $11 \text{ MPa}\cdot\text{m}^{1/2}$ . Since this level of fracture toughness is too low for most structural applications, this material could only be used in rare cases where application does not involve tensile stress.

Most nonaustenitic steels are likewise brittle at cryogenic temperatures, but a few are acceptable for service at 77 K. Acceptability is often judged by two fracture toughness criteria:

- 1) If a transition temperature is clearly identified, the steel must not be used at a service temperature lower than the transition temperature.
- 2) If a transition temperature is not obvious (as in high strength or high carbon steel), Charpy V-notch impact energy should be at least 21 J (15 ft·lbs) at the minimum service temperature.

On the basis of these conventions, the majority of ferrous metals are disqualified for NTF applications. As structural materials supporting tensile stresses, entire steel classes are unfit for service at 77 K, as listed in Table 4.1. Many steels favored for conventional wind tunnel testing (i.e., AISI 4340, and AISI 410) are unacceptable at 77 K. The acceptable steels for tensile applications are discussed in sections 4.2. and 4.3.

### 4.2. Nickel Alloy Steels

Nickel has beneficial effects on the cryogenic properties of steels. The beneficial effects include reduced ductile-to-brittle transition temperatures for ferritic steels, as well as higher toughness at subtransition temperatures. These effects are attributed to favorable influences on the deformation modes and cleavage strength of iron, combined with a tendency to promote the formation of retained austenite in some alloys. Taking into account the acceptability criteria of section 4.1, the following limited selection of nickel alloy steels is recommended as useful for NTF service:

- 1) low- to medium-strength austenitic Fe-Ni alloys
- 2) intermediate-strength, ferritic Fe-9Ni steel, and
- 3) high-strength, martensitic 18Ni maraging steel

All of these acceptable steels have low carbon contents, and high nickel content. Carbon contents higher than 0.2% harden the iron matrix and promote low temperature brittleness. Austenitic stainless steels containing Cr as well as Ni are also acceptable and are discussed in section 4.3.

Table 4.1. Suitability of ferrous alloys and steels for tensile structural applications at temperatures ranging from 367 to 77 K. (Examples are cited parenthetically).

Unacceptable	Acceptable
1. carbon steels (SAE 1005-1095)	1. austenitic Fe-Ni alloys (Fe-36Ni, Ni-Span C)
2. high strength low alloy steels (ASTM A94)	2. ferritic 9Ni steel (ASTM A353)
3. free machining steels (1109, 1151, 1211)	3. some 18Ni maraging steels (200 or 250 grades)
4. carburizing steels (3310, 4320, 8620)	4. various austenitic stainless steels (AISI 304, Fe-21Cr-6Ni-9Mn-0.3N)
5. direct hardening steels (3140, 4340)	
6. nitriding steels (N,135)	
7. low temperature carbon steels (ASTM A 514)	
8. tool steels (A2, A7, D1, D7)	
9. electrical steels (M4, M8)	
10. ferritic stainless steels (AISI 405, 430)	
11. martensitic stainless steels (AISI 410, 416)	
12. most ultrahigh-strength steels (300M, D6AC)	



## 4.2.1 Austenitic Fe-Ni Alloys

### a. General Information

Iron alloys with 36Ni or more have the austenitic (fcc) structure. Combined with low carbon content, this structure assures excellent ductility and toughness at low temperatures.

### b. Preferred Alloys

Invar (fcc structure) is a relatively low-strength alloy, best known for exceptionally low thermal contraction. Between 300 and 77 K, Invar contracts only 0.05%, whereas most metals contract 0.2 to 0.4%. The nominal composition is Fe-36Ni-0.1C-0.35Mn-0.3Si-0.1Al-0.015P-0.015S. The yield stress of Invar at room temperature is 289 MPa, only slightly higher compared to that of AISI 304. Invar is ductile, tough, and weldable. It can be used in the annealed or cold-worked conditions, and is recommended for applications where high strength is not required, but close dimensional stability is necessary to minimize thermoelastic stresses. Past cryogenic applications include measurement devices, glass window seals, bellows-less piping systems, and membrane liners for cryogenic storage tanks. This is the most widely used low expansion alloy.

Ni-Span C (fcc structure) is a medium-strength alloy that is recommended as a substitute for Invar in applications requiring higher strength and intermediate thermal contraction. Ni-Span C is a precipitation-hardenable alloy with a nominal composition: Fe-42Ni-0.06C(max)-5.2Cr-2.4Ti-0.5Al. Originally developed to achieve a constant Young's modulus between 227 and 338 K, Ni-Span C is often used for precision springs and bellows [60].

### c. Fabrication

Invar's high ductility and toughness render it as difficult to machine as austenitic stainless steel, but a modified grade (0.25e) for improved machinability is available. Annealing of Invar is performed by heating at 1073 K for half an hour per each 25 mm of thickness. A special three-step stability anneal for Invar confers minimum thermal expansion:

- 1) 1063 K, to 1103 K, 0.5 h per 25 mm, water quench,
- 2) 588 K, 1 h per 25 mm, air cool,
- 3) 368 K, 48 h, air cool.

Ni-Span C is solution treated and aged at 922 K for 5 h.

Weld procedures and joint designs are similar to those used for AISI 300 series stainless steels. Invar welds are compatible with stainless steel and nickel alloys. No preheating or postheating is required. Inconel 92 or Hastelloy W are general purpose fillers, but a modified Invar filler, Fe-36Ni-0.1C-3Mn-0.05Si(max)-1.0Ti-0.01S(max)-0.02P(max), more closely matches the base-metal expansion behavior.

### d. Availability

Invar is distributed by seven major corporations and is available in cast or wrought forms: strip, sheet, plate, wire, bar, tube, pipe, and billet forgings. Ni-Span C is less readily available. Costs for Invar and Ni-Span C are higher compared with stainless steels owing to higher nickel content.

### e. Mechanical and Physical Properties

Tensile: Between 300 and 77 K, the tensile properties of annealed Invar change as follows: yield stress increases from 289 to 620 MPa, ultimate strength increases from 552 to 862 MPa, and elongation decreases from 55 to 42%. For the same temperature interval the properties of heat-treated Ni-Span C vary as follows: yield stress increases from 128 to 1549 MPa, and elongation increases from 24 to 31% [10].

Charpy Impact: CVN values for Invar and Ni-Span C are high at all temperatures. A typical value for annealed Invar at 77 K is 65 J.

Elastic: Young's modulus for Invar is anomalous, decreasing from 152 to 139 GPa between 300 and 150 K, then increasing to 141 GPa at 77 K [61]. Data for Ni-Span C are not available.

Thermal Contraction: The thermal contraction of Ni-Span C between 293 and 80 K is 0.128%, compared with Invar's 0.05%. The linear thermal expansion coefficient for Invar is  $2.5 \times 10^{-6}/\text{K}$  at 293 K and  $0.83 \times 10^{-6}/\text{K}$  at 80 K, or about one tenth that of most steels.

Thermal Conductivity: The thermal conductivity of Invar decreases from 13.8 to 6.2 W/(m·K) between 367 and 77 K [11].

#### 4.2.2. Ferritic Fe-Ni Steels

##### a. General Information

From the finding that increased Ni content correlates with a progressive reduction of ductile-to-brittle transition temperatures, a family of low temperature ferritic steels was developed containing 2½ to 9Ni. The 9Ni grades are acceptable for NTF applications at 77 K, but all other grades in this family have insufficient nickel content and fail by cleavage at 77 K.

##### b. Preferred Alloys

Fe-9Ni is a medium-strength, weldable steel that has been extensively used in cryogenic applications at 77 K since 1947. In NTF structures, 9Ni steel is recommended as an alternative to AISI 304 stainless steels where a lower cost, higher yield strength, more machinable steel is desired. The nominal composition is Fe-9Ni-0.10C-0.6Mn. Two grades are available: ASTM A 353 (double-normalized and tempered) and ASTM A 553 Type I (quenched and tempered). The quenched and tempered grade offers 10% higher strength and slightly higher toughness.

##### c. Fabrication

The 9Ni steel is more easily machined compared with austenitic stainless steels, and is readily formed. Stress relief after cold forming is conducted at 824 to 857 K, for 2 h. Exposure at 643 to 813 K should be avoided or temper embrittlement will result. Heat treatments are performed by the supplier.

The 9Ni steel is weldable in 50-mm sections or less without preheating or postheating but there is no matching filler. Gas metal-arc welds use austenitic nickel base fillers (Inconel 82, 92, 182, or 625). Shielded metal arc welds use austenitic Inco Weld A or Inconel 112. Heat inputs are controlled to maintain adequate fusion and heat-affected zone properties. Welding is further described in product data bulletins. The mechanical properties of welds are summarized by Pense and Stout [62].

##### d. Availability

Several steel corporations produce 9Ni steel in the form of plates, forgings, pipe, tubing, bar sheet, or rolled sections. Plates are covered by the A 353 and A 553 specifications. Other forms are covered by ASTM specifications A 522, A 333, and A 334.

##### e. Mechanical and Physical Properties

Mechanical property data, including fatigue and fracture toughness data for ferritic Fe-Ni steels and welds, are compiled and summarized by Pense and Stout [62]. The properties quoted below are representative of A 553 Type I quenched and tempered 9Ni steel for temperatures decreasing from 300 to 77 K.

Tensile: Yield stress increases from 700 to 1000 MPa, ultimate strength increases from 800 to 1180 MPa, elongation is nearly constant at 24%, and reduction of area decreases from 67 to 57%.

Charpy: The CVN values decrease from 108 to 47.5 J for the transverse orientation.

Elastic: Young's modulus increases from 186 GPa to 207 GPa, and all of the elastic properties are reported to be regular functions of temperature [63].

Fracture Toughness: Fig. 3.5 illustrated the temperature dependence of fracture toughness for 9Ni steel. Typical  $K_{IC}$  estimates from J-integral tests are 156 to 205 MPa·m<sup>1/2</sup> at 295 K and 164 to 182 MPa·m<sup>1/2</sup> at 77 K [64-65].

Thermal Conductivity: The thermal conductivity of this steel decreases from 32 to 28 W/(m·K) between 373 and 295 K, and from 28 to 12.5 W/(m·K) between 295 and 73 K.

Thermal Contraction: The linear thermal expansion coefficient is  $10.4 \times 10^{-6}/K$  at 295 K and  $5 \times 10^{-6}/K$  at 73 K. Total contraction between 295 and 73 K is 0.35%. Expansion between 295 and 373 K is 0.15%.

#### 4.2.3 18Ni Maraging Steels

Ultrahigh-strength steels having nonaustenitic structures are infrequently used at cryogenic temperatures, since their toughness levels are lower than austenitic alloys. But a need for ultrahigh strength arises in some primary NTF components where the 77 K design stresses are projected to reach as high as 700 MPa. Maximum toughness at the required yield stress is paramount, and other factors, including cost, are secondary.

##### a. General Information

Available yield strength-versus- $K_{IC}$  data indicate that the 18Ni maraging steels offer superior strength and toughness combinations compared with other high strength steels, such as quenched and tempered 9Ni-4Co steels, AISI 4340, 300 M, or D6AC. Maraging steels have a martensitic structure, but the martensite is exceptionally tough owing to composition and low levels of impurities. At 77 K the 18Ni maraging steels remain tough owing to the effects of low carbon and high nickel contents. The 18Ni maraging steels also offer simpler heat treatments, deeper hardening with less distortion, and better dimensional stability on heat treating. Disadvantages are the lack of 77 K data and a growing problem of availability for maraging steels. Since fracture toughness decreases at cryogenic temperatures, accurate  $K_{IC}$  data and fatigue data are required, and additional materials testing must be performed at 77 K before these alloys can be safely used.

##### b. Preferred Alloys

The 18Ni maraging steels have the following compositional ranges: Fe-17 to 19Ni, 3 to 5.2Co, 0.15 to 2Ti, 0.05 to 0.2Al,  $\leq 0.03C$ ,  $\leq 0.10Mn$ ,  $\leq 0.01P$ . Four grades are available, designated 200, 250, 300, and 350, which indicates the nominal yield stress in ksi at room temperature. Specifications covering these steels are MIL-S-46850 (Mi) and Aerospace Materials Documents specifications 64BL, 64BM, 64BN, 64BP-1, and 64BR-1. To date the 18Ni maraging steels have seen limited cryogenic service, having been used as specimen grips and load frame components for cryostats at 77 K or lower. The 200 and 250 grades are recommended for NTF applications, but the 300 and 350 grades are not recommended in view of lower cryogenic impact and toughness values.

##### c. Fabrication

The 18Ni maraging steels are stronger than austenitic superalloys or stainless steels, and yet easier to fabricate. Machining in the annealed condition is comparable to that of AISI 4340 steel at similar hardness, R<sub>30</sub>. After air cooling from the solution annealing temperature, 1088 K, maraging steels are hardened in a single 3 h aging cycle at 755 K. No protective atmosphere is needed, just air, and there is no decarburization problem since the initial carbon content is low.



In welding maraging steels the GTA process is preferred, with techniques similar to stainless steels. Preheating or postheating is not recommended. Joint efficiencies of 90% can be expected using matching fillers. Best properties are achieved by welding in the annealed condition.

#### d. Availability

The 18Ni maraging steels are furnished as plate, sheet, billet, rod, and bar, in the mill-annealed condition. Bar is the most common form, but not all forms are available from every supplier, and availability is decreasing. Costs are high and vary with grade, size, and quantity desired.

#### e. Mechanical and Physical Properties

The properties of 18Ni maraging steels have not been extensively characterized at low temperatures. Some available mechanical properties for three grades are listed in Table 4.2. The 200 grade maraging steel is perhaps the best candidate, showing an increase in yield stress from 1418 to 1861 MPa at temperatures between 295 and 77 K, in combination with high ductility, impact and toughness values.

In addition to the data of Table 4.2, Shoemaker and Rolfe [66] and Clausing [67] report yield stress and fracture toughness data for 200 and 250 grade maraging steels. Between 295 and 77 K,  $K_{IC}$  for the 250 grade decreased linearly from 110 to 44 MPa·m<sup>1/2</sup>. The  $K_{IC}$  value for the 200 grade steel at 77 K was 126 MPa·m<sup>1/2</sup>. Nachtigall [25] and Tobler, Reed, and Schramm [68] report cryogenic fatigue data for 300 grade steels.

#### 4.2.4. Other Ferrous Alloys

Maraging steels containing 20Ni or 25Ni were originally produced in experimental quantities but they offered no advantages compared with the 18Ni grades and so have fallen from favor and availability. More recently 12Ni-5Cr-3Mo maraging steels have been developed but cryogenic data are unavailable.

The quenched and tempered 9Ni-4Co series steels containing 0.20 to 0.45% carbon are probably the closest competitors to 18Ni maraging steels for high strength applications. The higher carbon grades, such as 9Ni-4Co-0.45C, exhibit inferior impact toughness at 77 K. The lowest carbon grade, 9Ni-4Co-0.20C, covered by AMS-6523 and AMD 62 CF-1 specifications, is more attractive. The reduction of area for this grade is a respectable 62% at 300 K and 51% at 77 K, and the yield stresses at these temperatures are 1265 MPa and 1654 MPa respectively. Nevertheless, Vishnevsky and Steigerwald [7] indicate that 200 grade 18Ni maraging steel offers superior strength and fracture toughness, as well as simpler fabrication.

A cast iron that Pearson [69] considers acceptable at 77 K is Ni-resist cast iron type D-2M. The composition of this material is Fe-2.9C-22Ni-4Mn-2.5Si. The yield stress at 295 K is 231 MPa. However, cryogenic property data are not available.

Another alternative to consider is PH 13-8Mo or related alloys (section 4.3.5). PH 13-8 Mo in the H 1150-M condition offers a yield stress of 583 MPa at 295 K, or 993 MPa at 77 K, with a 50% reduction of area at 77 K [7]. Unfortunately, valid fracture toughness data are lacking for alloys of this type.

#### 4.3. Stainless Steels

Steels containing 12-30% chromium to resist corrosion are classified as stainless steels [70]. There are five commercial alloy systems:

- 1) AISI 200 series (austenitic Fe-Cr-Mn-Ni alloys)
- 2) AISI 300 series (austenitic Fe-Cr-Ni alloys)
- 3) AISI 400 series (ferritic or martensitic Fe-Cr alloys)

Table 4.2. Mechanical properties of aged 18Ni maraging steels.

Material	Test temperature K	Yield stress MPa	Ultimate strength MPa	Red. of area %	Young's modulus GPa	Poisson's ratio	Charpy impact energy J	Fracture toughness, $K_{Ic}$ MPa·m <sup>1/2</sup>
200 grade	295	1418	1461	65	193.7	0.311	49	187
	77	1861	1930	60	203.0	0.306	39	86
250 grade	295	1696	1792	--	195.3	0.308	28	110
	77	2206	2275	--	204.8	0.304	14	44
300 grade	295	1903	1982	55	196.8	0.308	25	--
	77	2344	2475	40	206.3	0.303	12	--

Note: 1 MPa = 0.145 ksi  
1 GPa = 0.145 msi  
1 J = 0.737 ft. lb  
1 MPa·m<sup>1/2</sup> = 0.91 ksi

- 4) nitrogen-bearing series (austenitic Fe-Cr-Ni-Mn-N alloys)
- 5) AISI 600 series (various precipitation hardening alloys).

The AISI 300 series stainless steels are the most widely used cryogenic alloys.

#### a. General Information

AISI 200 series (austenitic Fe-Cr-Mn-Ni alloys). The 200 series stainless steels are not recommended for cryogenic service. These alloys were developed for room temperature service as substitutes for the 300 series steels at a time when nickel was difficult to obtain. Compared to the 300 series stainless steels, the 200 series steels contain less Ni and more Mn; they are less stable with respect to martensitic transformation and less ductile at 77 K.

AISI 300 series (austenitic Fe-Cr-Ni alloys). By far the most widely used cryogenic alloys are the AISI 300 series stainless steels. These steels contain 16-26% Cr, 6-22% Ni, not more than 2% Mn, and various other elements in smaller quantities. Nickel tends to stabilize the austenitic crystal structure, but not all grades in this series retain a fully austenitic structure at low temperatures; small amounts of martensite spontaneously form in some grades after cooling to 77 K (section 3.8).

In addition to excellent cryogenic properties, the 300 series stainless steels offer ease of fabrication, extensive service history, and ready availability in all standard product forms and sizes. The main disadvantages are: (1) higher material and machining costs compared with aluminium alloys or ferritic 9Ni steel, and (2) complications associated with sensitization or martensitic transformation in some grades.

The 300 series stainless steels are typically used in the annealed condition, with yield stresses not exceeding 275 MPa at room temperature. Nitrogenated "N" grades can be used where higher strength and austenite stability is required, but availability and service experience is limited for N grades. Cold working is another satisfactory method of gaining strength, but only for thin sheet.

Sensitization is a problem encountered when stainless steels are heated at 700 to 1088 K for significant time periods. At these temperatures, chromium carbides precipitate at grain boundaries, ruining corrosion resistance, reducing ductility and toughness, and lowering stability with respect to martensitic transformation. Sensitization frequently occurs in the heat-affected zones of welds, for example, and there are two methods of combating it. One approach is to select stabilized grades such as AISI 321 or 347 that contain Cb and Ta additions that form carbides in preference to Cr. The second approach is to select low carbon grades designated "L" or "S". The low carbon grades have lower yield strengths, but their welds offer higher impact resistance compared with welds of stabilized Cb/Ta grades.

AISI 400 series (ferritic or martensitic Fe-Cr alloys). Owing to unfavorable crystal structures these steels are hard, abrasion resistant, and brittle at cryogenic temperatures. AISI 440-C steel has been used as a ball bearing alloy at 77 K; but otherwise none of the 400 series steels can be recommended for cryogenic service.

Nitrogen-bearing series (austenitic Fe-Cr-Ni-Mn-N alloys). The addition of manganese to austenitic stainless steels helps to stabilize the austenitic structure and increase the solubility limit for nitrogen, which significantly increases the yield stress. Two types of nitrogen-strengthened stainless steels are available: the N-grade AISI 300 series alloys, or the Fe-Cr-Ni-Mn-N alloys considered here. The Fe-Cr-Ni-Mn-N alloys typically contain 0.3N, whereas AISI 304N contains 0.10 to 0.16N. There are five alloys in the relatively new Fe-Cr-Ni-Mn-N series:

- 1) Fe-18Cr-2Ni-12Mn-0.3N
- 2) Fe-18Cr-3Ni-13Mn-0.3N
- 3) Fe-21Cr-6Ni-9Mn-0.3N
- 4) Fe-21Cr-13Ni-5Mn-0.3N, and
- 5) Fe-17Cr-8Ni-8Mn-0.3N



In the annealed condition in which they are used, these alloys offer yield stresses at least 25% greater than AISI 304N. The preferred grade for cryogenic service is Fe-21Cr-6Ni-9Mn-0.3N, because of its relatively high stability, more extensive data base, and good properties at 77 K. Disadvantages are higher cost and limited availability and service experience compared to AISI 300 series steels.

AISI 600 series (various precipitation-hardening alloys). There are more than a dozen steels in this series, classified as martensitic, semiaustenitic, or fully austenitic. Examples of martensitic or semiaustenitic grades are stainless W(AISI 635), 17-4 PH (AISI 630), 15-5 PH, PH 13-8Mo, 17-7 PH (AISI 631), 15-7 PH (AISI 632), AM 350 (AISI 633), and AM 355 (AISI 634). These steels are too brittle in the fully aged conditions for general applications at 77 K. Therefore, the H temper conditions are mandatory. Even so, cryogenic applications are rarely recommended. Alloy 15-5 PH in the H 1150 M temper has been used for valve seats at 77 K, but the CVN values are marginal, ranging from 6.7 to 38 J.

Most of the fully austenitic grades also have inferior cryogenic properties, but one alloy, A-286, is acceptable for cryogenic service [60]. Alloy A-286 offers the advantage of a higher yield stress in the precipitation-hardened condition, compared with annealed AISI 300 series steels. Disadvantages are high cost, and more difficult fabrication than the 300 series steels.

#### b. Preferred Alloys

From the information above, the preferred stainless steels for NTF service are several steels from the AISI 300 series, Fe-21Cr-6Ni-9Mn-0.3N, and A 286.

AISI 302 (Fe-18Cr-8Ni<0.15C) is the basic 18-8 stainless steel, the usual choice for room temperature service. But AISI 304 (Fe-18Cr-8Ni<0.08C) is preferred for cryogenic service since it has lower carbon content. AISI 304 was chosen for construction of the NTF wind tunnel walls.

There are about two dozen steels in the AISI 300 series, each developed for a specific purpose [72]. Most grades having more than 0.08% carbon are not used at 77 K, but an exception is AISI 301 which has been used as high-strength, cold-worked sheet for aerospace skin structures at 77 K. More commonly used at cryogenic temperatures are AISI 304, 304L, 316, 310S, and the sensitization resistant grades 321 and 347.

AISI 304L (Fe-18Cr-8Ni<0.03C) has lower carbon than AISI 304 and is substituted for welded structures to further reduce sensitization. AISI 316 and 310S are more highly alloyed than either 304 or 304L and are more resistant to martensitic transformation. In fact, AISI 310S (Fe-25Cr-20Ni<0.03C) is completely stable and forms no martensite when fractured at cryogenic temperatures. Specifications for the austenitic stainless steels are listed in Table 4.3. The federal specifications for AISI 300 series stainless steels are QQ-S-766 and QQ-S-763.

Alloy Fe-21Cr-6Ni-9Mn-0.3N is covered by ASTM specifications A 412 for plate, sheet, and strip and A 276 for bars and pipes. The alloy has a relatively high austenite stability compared with AISI 304, and the yield stress at 300 K is about 353 MPa, compared with 200 MPa for AISI 304. Cryogenic applications to date include torque tubes and rotors for superconducting machinery at temperatures as low as 4 K.

Alloy A 286 has the nominal composition Fe-25Ni-14Cr-2.2Ti-1.5Mn-1.2 Mo-0.3V-0.2Al-0.5Si. Considerable cryogenic data exist for this alloy, which is nonmagnetic and completely stable with respect to martensitic transformation when cooled or stressed at cryogenic temperatures. The yield stress in the fully hardened condition at 300 K is 550-750 MPa. This alloy is recommended for applications requiring a stable fcc structure and significantly higher yield stress than any of the annealed austenitic stainless steels, particularly at room and elevated temperatures.

Table 4.3. ASTM and ASME specifications for various forms of austenitic stainless steels.

Product Form	ASTM specifications	ASME specifications
Plates	A240	SA-240
Forgings	A473	SA-182
Bars and sections	A479	SA-479
Seamless tubes	A213	SA-213
Welded tubes	A249	SA-249
Seamless and welded tubes	A269	--
Bolts, screws, studs, stud bolts	A320	SA-320
Nuts for bolts	A194	SA-194

### c. Fabrication

The high ductility of austenitic stainless steels insures good formability, but greater force is needed compared with aluminum alloys or plain carbon steels. The austenitic steels can be formed by bending, cold-roll forming, tube forming, drawing, and spinning, but difficulty increases with increasing yield stress. Alloy A-286 is particularly difficult to form and machine.

Casting alloys are available and may be used if complex shapes are needed, but cast products have inferior mechanical properties compared with wrought products. Casting alloy CF-8 is the analog of the wrought alloy AISI 304. For information on cast stainless steels, consult Peckner and Bernstein [70]. Forging is also possible, but austenitic stainless steels are generally more difficult to forge than most structural alloys owing to their relatively high strength at elevated temperatures.

High work hardening and low thermal conductivity make annealed stainless steels more difficult to machine than plain carbon steels. Selection of cutting fluids is important to extend tool life by lubrication. It is necessary to use heavy feeds, slow speeds, high quality tool bits, and rigid machines. Guides to machining are available in technical notes from suppliers.

The austenitic stainless steels are annealed at temperatures between 1227 and 1422 K, depending on composition, or stress relieved at 477 to 700 K. Stress relieving treatments reduce residual stress or improve dimensional stability. Rapid cooling is specified because the interval 700 to 1088 K must be avoided to prevent sensitization, and the interval 1005 to 1162 K must be avoided to prevent formation of brittle sigma phase. Alloy A-286 is usually solution treated (1172 K) and aged (1005 K, 16 h, air cooled).

The austenitic stainless steels can be soldered using Pb-Sn solders and special fluxes to break down oxide films. They can also be brazed. Brazing is conducted at 1255 to 1366 K, followed by rapid quench. Alloy A-286 is preferably brazed in vacuum, with nickel plating to improve wettability.

Except for AISI 303, all austenitic stainless steels are considered weldable [71]. Gas tungsten arc welding is preferred for flat stock, while shielded metal arc is preferred for gages thinner than 1 mm. Oxyacetylene, electron beam, or resistance welding techniques are also possible. Among the problems encountered in welding are sensitization and hot cracking. To minimize sensitization, stabilized grades (AISI 321, 347, 348) and AISI 304L or 308L fillers are often preferred. Fillers are formulated to provide 4 to 10% delta ferrite to reduce hot cracking.

For alloy A 286, available fillers are Inconel 92, Hastelloy W, and AISI 310, in addition to matching fillers. For Fe-21Cr-6Ni-9Mn-0.3N, type 308 filler can be used, but it offers lower strength compared with the base metal. N 40W is a matching filler for Fe-21Cr-6Ni-9Mn-0.3N, which gives good cryogenic toughness in sections up to 6 mm; Inconel-type fillers are preferred for thicker sections.

### d. Availability

The availability of AISI 304, 316, 310 and alloy Fe-21Cr-6Ni-9Mn-0.3N is unlimited in standard product forms, including sheet, strip, wire, bar, plate, tubing, and forging billets. Costs for alloys in bar form range up to \$2.3/kg. Costs vary considerably with form and increase with higher alloy content. Alloy A-286 is available as vacuum arc remelted billets, bars, and wire, in the solution-treated or aged conditions; but not every supplier furnishes all forms, and costs are higher than AISI 300 series steels.

### e. Mechanical and Physical Properties

Brickner and Defilippi [72] and Sanderson and Llewellyn [73] review the mechanical properties of austenitic stainless steels. Extensive mechanical and physical property data for the recommended stainless steels are available in handbooks and reference books [74].



**Tensile:** The yield stress of annealed AISI 300 series stainless steels range from 172 to 262 MPa at 77 K. Among AISI 304, 310, and 316, AISI 304 has the lowest yield strength at 77 K (owing to lower alloy content), but the highest ultimate strength (due to greater strain-induced martensite content). AISI 321 and 347 behave similarly to AISI 304. Data for high strength alternatives including AISI 304N, Fe-21Cr-6Ni-9Mn-0.3N, and A-286 are shown in Fig. 4.1. Using these alloys, 77 K yield strengths of 399 MPa, 353 MPa, and 550-750 MPa can be achieved expense of ductility.

Tensile data for cold worked AISI 301, 304, 304L, and 310 stainless steel sheet are also available at cryogenic temperatures [75]. The effect of cold-work is to increase strength at the expense of ductility. Cold-work is usually indicated by the terms quarter, half, or full hard. Data for AISI 310 are reported by Christian, Gruner, and Girton [76] show a 300 K yield stress of 1158 MPa at 80% cold-work, compared to 278 MPa for the annealed condition. But elongation for the 80% cold-worked condition is only 2% at 300 K.

**Elastic:** For AISI 304 stainless steel, Young's modulus is 1.896 GPa at 300 K. The 77 K amounts to 8%. The effect of temperature between 295 and 77 K is not always regular, but may be anomalous for some austenitic stainless steels, as explained in section 3.4 [77-78].

**Impact:** Charpy impact values for the recommended stainless steels are high at all temperatures. In general there is a modest reduction of CVN between 300 and 77 K. Representative CVN data for AISI 304 are 150-217 J at 295 K and 122-150 J at 77 K.

**Fatigue:** Stress-controlled tests show improved fatigue strengths and high fatigue resistance for smooth bars and notched bars between 295 and 77 K. Fatigue crack growth rates for AISI 310 and A286 decrease between 295 and 77 K, while for AISI 304 and AISI 316 there is little change in rates measured at these two temperatures [31].

**Fracture Toughness:** Annealed austenitic stainless steels exhibit ductile fractures and high toughness, even when deeply cracked. The toughness of annealed alloys is so high that J-integral methods are necessary to estimate  $K_{IC}$  values [79-82]. Representative results for AISI 310S are shown in Fig. 3.5, where toughness estimates increased to  $160 \text{ MPa}\cdot\text{m}^{1/2}$  at 77 K. The toughness-to-yield strength ratios for annealed austenitic stainless steels are such that significant plastic deformation is expected to precede fracture and a fracture mechanics analysis is generally not required. Fracture toughness decreases for highly cold-worked, precipitation-hardened, or welded and sensitized steels. For example, alloy A-286, in the precipitation-hardened condition indicates relatively low toughness values of about 114 to  $161 \text{ MPa}\cdot\text{m}^{1/2}$  at 295 K. Since material variability is significant at these lower toughness values, quality control and fracture mechanics analysis may be appropriate when this alloy is used in critical applications.

**Thermal Conductivity:** The thermal conductivity of AISI 304 decreases from  $16 \text{ W}/(\text{m}\cdot\text{K})$  at 295 K to  $8.2 \text{ W}/(\text{m}\cdot\text{K})$  at 77 K. The values for other stainless steels are indicated in Fig. 3.12.

**Thermal Expansion:** Thermal expansion coefficients and total contractions for austenitic stainless steels are reported by several investigators [44-47]. The total contraction for the AISI 300 series steels between 367 and 90 K is typically 0.4%; contraction between 293 and 80 K is about 0.28%.

#### 4.4 Aluminum and Its Alloys

Aluminium and its alloys (fcc structure) are excellent cryogenic materials whose mechanical and physical properties are regular, predictable functions of temperature [83]. Pure aluminum offers high thermal and electrical conductivities. Aluminum alloys offer high strength-to-weight ratios, low costs, and ease of fabrication. Disadvantages are

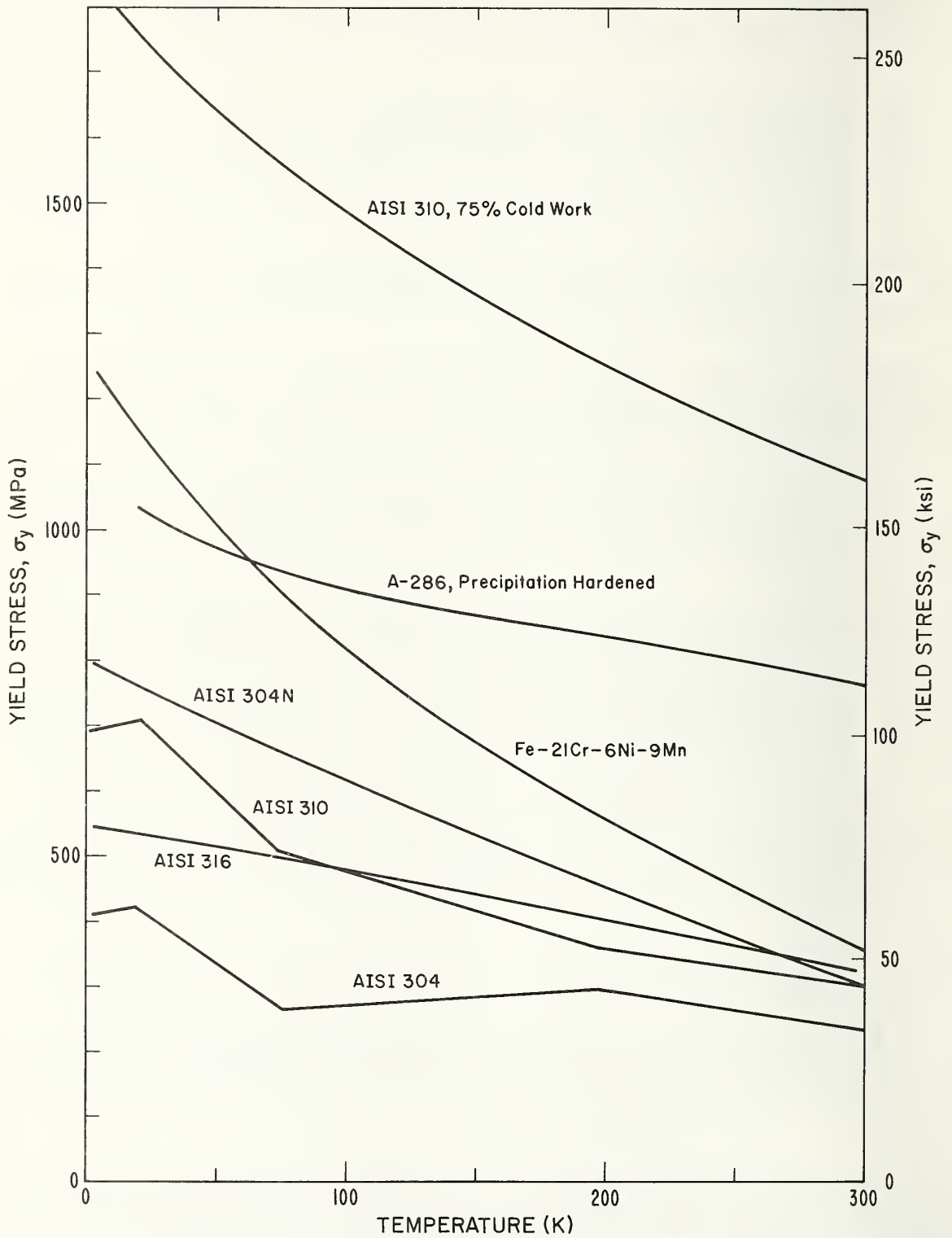


Figure 4.1. Yield stress data for stainless steels.

higher thermal contraction, lower strength, and lower stiffness than copper, titanium, and steel alloys.

#### a. General Information

Wrought aluminum alloys are classified by four-digit numbers followed by temper designations. The alloy families are:

- 1000 series - unalloyed aluminum
- 2000 series - aluminum/copper
- 3000 series - aluminum/manganese
- 4000 series - aluminum/silicon
- 5000 series - aluminum/magnesium
- 6000 series - aluminum/magnesium/silicon
- 7000 series - aluminum/zinc

Temper designations are 0, H, or T to denote the annealed, cold-worked, and aged or heat-treated conditions, respectively. The 2000, 6000, 7000, and some 4000 series alloys are precipitation hardenable. Other alloys can be cold-worked to improve strengths moderately.

#### b. Preferred Alloys

Most aluminum alloys are acceptable for cryogenic applications [84]. Most commonly used are the 5083-0, 6061-T6, and 2219-T8 alloys. All three of these are readily welded, but only 5083-0 alloy can be welded to a joint strength efficiency of 100%. Specifications for these alloys are listed in Table 4.4.

Alloy 5083-0 has the nominal composition Al-4.5Mg-0.6Mn. It is used in massive quantities as a primary structural alloy for large cryogenic storage tanks. It can be cold-worked but is typically used in the annealed condition for maximum ductility and toughness. Alloy 5083-0 has a 300 K yield stress of 140 MPa.

Alloy 6061-T6 has the nominal composition Al-1Mg-0.6Si-0.27Cu-0.2Cr. This alloy is widely used in cryogenic piping systems, offering intermediate strength and ductility, with a 300 K yield stress of 270 MPa.

Alloy 2219 has the nominal composition Al-6.3Cu-0.3Mn. In the T87 temper this alloy offers high strength, moderate ductility, and adequate fracture toughness for weight-saving applications. The 300 K yield stress is 388 MPa. Applications for alloy 2219 include airborne cryogenic pressure vessels.

Many other aluminum alloys are also useful for specific purposes. Alloys 2024 and 7075 are recommended for nuts and bolts or bolted components that do not require welding. Unalloyed aluminum EC is a conductor grade rivaling copper in cost. Commercial purity 1100-0 aluminum offers superior formability and corrosion resistance. Alloy 3003 is used for heat exchangers and is the most brazable aluminum alloy. Alloy A712 offers the highest ductility of any casting alloy at 77K. Alloy 7005-T5351 offers the best combination of weldability, strength, and toughness of any 7000 series alloy. Alloy 2021 offers the highest strength possible in the aluminum system but is the least ductile. Alloy 2014 is stronger than 2219, but less weldable.

#### c. Fabrication

Aluminum alloys are among the easiest alloys to form, forge, cast, and machine. Pure aluminum is soft and gummy when machined, but heat-treated alloys have such excellent machinability that machined and bolted structures of high strength aluminum alloys are often more economical to produce than welded stainless steels. Heat treatment is usually performed by the producer. Solution-treatment temperatures are 715 to 750 K for the 7000 series alloys and 760 to 810 K for the 2000 or 6000 series alloys.

Table 4.4. Specifications for aluminum mill products.

Alloy	Product form	Federal	ASTM	ANSI
5083	Sheet and plate	QQ-A-250/6	B209	H38.2
	Wire, rod, bar, shapes, tube	QQ-A-200/4	B221	H38.5
	Forgings and forging stork	QQ-A-367	B247	H38.8
2219	Sheet and plate	QQ-A-250/30	B209	H38.2
	Wire, rod, bar: rolled or cold finished	--	B211	H38.4
	Wire, rod, bar: extruded	--	B221	H38.5
	Forgings and forging stork	QQ-A-367	B247	H38.8
6061	Sheet and plate	QQ-A-250/11	B209	H38.2
	Wire, rod, bar: rolled or cold	QQ-A-225/8	B211	H38.4
	Wire, rod, bar, shapes, tube	QQ-A-200/8	B221	H38.5
	Structural shapes	QQ-A-200/16	B308	H38.10
	Pipe	--	B241	H38.7
	Forgings and forging stork	QQ-A-367	B247	H38.8
	Structural pipe and tube: extruded	--	B249	H38.16



Gas metal arc and gas tungsten arc are the preferred welding processes. Alloy 5083-0 retains full strength in the as-welded condition, and with a tensile strength of 140 MPa at 300 K, 5083-0 welds offer higher strength than 6061 or 2219 welds unless the latter are heat treated. Postweld heat treatment can restore the strength of 2219 welds to nearly 100% of base metal values.

#### d. Availability

Most alloys including 5083, 6061, and 2219, alloys are available as plate, sheet, strip, bar, and forgings. Newly developed alloys, such as 7005, are difficult to obtain except as forgings and extrusions. Alloy 6061 is readily available in pipe form or related geometries. The cost of a 2.5-cm thick aluminum alloy plate is about \$2/kg. Costs do not vary as widely for the different grades as they do for stainless steels.

#### e. Mechanical and Physical Properties

The cryogenic properties of aluminum are regular functions of temperature and stereotypic of an fcc system. The temperature reduction from 300 to 77 K is favorable, resulting in moderately improved tensile, fracture, and fatigue properties.

**Tensile:** Yield stresses at 300 K range from 48 MPa for unalloyed 1100-0 aluminum to 459 MPa for 2021-T815 alloy. Between 300 and 76 K, 1100-0 aluminum shows a yield stress increase from 48 to 62 MPa, a tensile strength increase from 93 to 191 MPa, an elongation increase from 46 to 52%, and a reduction of area decrease from 88 to 81%. Similar trends are observed for aluminum alloys.

**Elastic:** Young's modulus for unalloyed aluminum is 69.4 GPa at 300 K. Alloys have higher values, typically 70 to 78 GPa [85-86]. The increase between 300 and 77 K is typically 8%. The lower elastic moduli of aluminum alloys compared with steel causes a factor of three greater elastic deflections and 1/3 lower buckling resistance compared with similar-sized components of stainless steel.

**Impact:** There is usually little change and sometimes a modest increase of impact resistance at lower temperatures.

**Fatigue:** The fatigue resistance of aluminum alloys at room temperature is sensitive to ambient moisture. Crack initiation and propagation occurs more readily in humid conditions. Fatigue crack growth rates at 77 K are significantly lower than at 300 K [87-89]. The notch sensitivity in conventional S-N tests does not increase at lower temperatures. Strain cycling tests on 2014-T6 and 2219-T851 show cross-over behavior, where cycle life is higher at 77 K for lives longer than 500 cycles and higher at 300 K for lives longer than 500 cycles [25].

**Fracture Toughness:** Annealed alloys (5083-0) fracture by ductile tearing with high toughness at all temperatures. The high strength alloys (2219-T8) exhibit linear-elastic fast fractures, but seldom is there a large decrease in toughness between 300 and 77 K. The  $K_{IC}$  values for precipitation-hardened 2000 series alloys (2014, 2024, 2021, and 2219) range from 20 to 30  $\text{MPa}\cdot\text{m}^{1/2}$  at 300 K, and there is typically a 20% increase at 77 K [89-90].

**Electrical Resistivity:** The electrical conductivity of EC grade aluminum at 300 K is 62% IACS. The resistivity dependence on alloying (shown in Fig. 3.8) is discussed by Clark [38].

**Thermal Conductivity:** The thermal conductivity of 1100-0 aluminum is 210  $\text{W}/(\text{m}\cdot\text{K})$  at 300 K and 280  $\text{W}/(\text{m}\cdot\text{K})$  at 77 K. Structural alloys including 2014-T6, 2219-T81, 5083-0, 6063-T5 and 7075-T6 have lower values, 100 to 180  $\text{W}/(\text{m}\cdot\text{K})$  and 50 to 130  $\text{W}/(\text{m}\cdot\text{K})$  at 77 K. Thus, cryogenic temperatures improve the thermal conductivity of unalloyed aluminum but decrease the conductivity of alloys.

Thermal Expansion: The linear thermal expansion coefficients for aluminums range between  $20 \text{ and } 24 \times 10^{-6}/\text{K}$  at 300 K and between  $8.5 \text{ and } 10 \times 10^{-6}/\text{K}$  at 77 K. Total contraction for this temperature interval is typically 0.35 to 0.41%, which is higher than other structural metals.

#### 4.5 Copper and Its Alloys

Unalloyed copper (fcc structure) offers higher electrical and thermal conductivities than other metals. Copper alloys offer medium strength and modulus, easy fabrication, and convenient joining by soldering or brazing. High costs preclude the use of copper in large quantities, but practical applications exist for small components, mainly in secondary structures where fabrication or physical property advantages are paramount.

##### a. General Information

Copper and its alloys are conventionally specified using the three-digit designations. The alloy systems of primary interest are unalloyed coppers, brasses, bronzes, precipitation-hardening coppers, and cupronickels.

There are only two precipitation-hardening alloys: Pd-135 and beryllium coppers. Other copper alloys must be strengthened by solid solution hardening or cold work. Every method of strengthening compromises thermal and electrical conductivities. Thus, there are no high-strength, high-conductivity copper alloys. This fact, combined with rising materials costs and higher stress requirements has generally forced copper alloys out in favor of less expensive aluminum or ferrous alloys that offer competitive strength, machinability and conductivities in many cases.

##### b. Preferred Alloys

Large quantities of pure copper are sold for electrical applications. Unalloyed coppers (100 series) have the highest electrical and thermal conductivities, but they are soft. NTF applications would use these conductor grades for electrical wiring. The electrical conductivities of grades 101 and 110 are rated 100% by the International Annealed Copper Standard (IACS). These commercial grades are better electrical conductors than aluminum. Most widely used for electrical wiring is electrolytic tough pitch (ETP) copper 110 (99.9%Cu). Oxygen free copper (CDA 101, 99.99%Cu) has even higher purity and conductivity. The yield stress of copper 101 at 300 K is 75 MPa.

Pd-135 is a Cu-0.3Cd-0.3Cr alloy, which offers improved strength compared with unalloyed copper at a modest sacrifice of conductivity. In the precipitation-hardened condition, the yield stress at 300 K is 255 MPa and electrical conductivity is 90% IACS. Possible uses for Pd-135 include high tensile conductor wire or electrical contacts.

Beryllium coppers (grades 170, 172, 173) are based on the Cu-1.8Be composition, with some additions of cobalt. These precipitation-hardening alloys offer the highest strengths in the copper system, with 300 K yield stress in the aged condition reaching 1000 MPa, or 18 times the value of unalloyed, annealed copper. However, the electrical conductivity of beryllium coppers is only 4-10% IACS. In cryogenic service, beryllium copper has been used for springs and other special items.

Medium-strength brasses and bronzes are routinely used for small, machinable components in cryogenic apparatus. Bronze is a broad term covering alloys in the 500 and 600 series. Phosphor, silicon, and aluminum bronzes offer corrosion resistance, weldability, and good mechanical strength. Brasses are Cu-Zn alloys, available in three structural types:

- 1) Alpha brasses (fcc, less than 39Zn)
- 2) Alpha + beta brasses (fcc + bcc, 39 to 46.6Zn)
- 3) Beta brasses (bcc, 46.6 to 50Zn)



The beta brasses, unlike other bcc metals, are not brittle at cryogenic temperatures. Nevertheless, the alpha brasses are more widely favored for cryogenic service. Plain Cu-Zn brasses are numbered in the 200 series. Leaded brasses having Pb additions for improved machinability are numbered in the 300 series. The 360 grade is most popular for machining. Brass having Sn additions to improve strength are numbered in the 400 series.

The cupronickels (between 5 and 45% Ni, some Fe, and some Mn) offer corrosion resistance and weldability. Nickel silvers (60-65% Cu, 7-30% Ni, and the balance Zn) constitute an alternative to stainless steel for tubing. Constantan is a generic name for the Cu-40% Ni alloys, which find uses in heater windings and thermocouples by virtue of their high temperature insensitive electrical resistance.

#### c. Fabrication

The cold formability of copper and its alloys is excellent. The best cold-forming grades have a single-phase fcc structure: solution annealed beryllium copper, 70/30 brass, and unalloyed conductor grades 101, 110. The two-phase alloys, such as alpha/beta brasses, are preferable for hot working.

Coppers are nearly as easy to machine as aluminum alloys. On a relative scale, free cutting leaded brass grade 360 ranks 100% machinable, whereas unleaded brass ranks 30%. Least machinable are the soft conductor grades 101, 110, which work-harden significantly.

The precipitation treatment for beryllium coppers consists of a 1- to 3-h heating at temperatures ranging from 588 to 673 K. Other alloys are typically annealed at temperatures ranging from 588 to 977 K.

Copper and its alloys are readily joined by soft soldering. Brazing is also readily accomplished, noting precautions. Brazing temperatures will anneal cold-worked base metals. Therefore, temperature control is needed in critical joining operations where filler melting and base metal annealing points are close. Certain fluxes must be used to prevent dezincification or loss of Pb in leaded brass. Welding usually involves the gas tungsten-arc process for materials less than 4.8-mm (0.187-in) thick, and the gas metal-arc process is best suited for thicker gages.

#### d. Availability

There are few limitations on availability of copper. The wide variety of product forms reflects the high formability of copper alloys: wire, tubing, valves, springs, screws, gaskets, fasteners, seamless pipe, sheet, rods, plates, and bars. Cost is the more severe restriction. Copper is typically twice as expensive as AISI 304 stainless steel. Copper wire products costs 2 to 8 times higher than aluminum wire, depending on purity of grades. Copper costs are significantly reduced, however, using alloys such as brass with sizable additions of zinc.

#### e. Mechanical and Physical Properties

Low temperature effects in the copper systems are qualitatively similar to those in the aluminum system. In terms of mechanical behavior, improvement or little change is expected in tensile, fatigue, and impact behavior as temperature is reduced between 295 and 77 K. Cryogenic data are compiled in the monograph by Reed and Mikesell [91].

**Tensile:** Between 300 and 76 K, annealed oxygen-free copper, exhibits a stress of about 66 to 89 MPa with elongations of 53 to 60%. Over the same range, beryllium copper, the strongest copper, exhibits a yield stress of 985 to 1200 MPa with elongations of 6 to 10% [92]. Cartridge brass is representative of the majority of copper alloys, which achieve intermediate strength and ductility as a result of solid solution hardening and cold work.

**Elastic:** Young's modulus for copper at room temperature is 129 GPa, slightly higher than values for titanium. The moduli for copper alloys at 295 K varies with composition, but the temperature dependence between 300 and 77 K is regular, increasing by about 7 or 8% [93-94].

Charpy Impact: CVN values usually increase or remain nearly constant at cryogenic temperatures. For example, CVN data for annealed oxygen-free copper between 300 and 77 K increase from 71 to 89 J. Aged beryllium copper increases from 6.6 to 9.5 J.

Fracture Toughness: Copper alloys are not commonly used for fracture-critical components. Therefore,  $K_{IC}$  data are unavailable. Brittle fracture is possible for heavily cold-worked alloys or for aged beryllium copper. Alloys in the annealed condition are highly ductile and fracture by gradual tearing [95].

Electrical Resistivity: The electrical resistivity of oxygen-free copper is sensitive to purity, but representative values are  $1.56 \mu\Omega\text{cm}$  at 273 K and  $0.2 \mu\Omega\text{cm}$  at 76 K. Higher resistivities and regular temperature dependences are observed for alloys [38].

Thermal Conductivity: The 300 K thermal conductivities are highest for oxygen-free copper  $400 \text{ W}/(\text{m}\cdot\text{K})$  and lowest for German silver, Manganin, and Constantan: 22 to  $25 \text{ W}/(\text{m}\cdot\text{K})$ . A temperature reduction to 77 K improves the conductivity of oxygen-free copper to  $550 \text{ W}/(\text{m}\cdot\text{K})$ , but decreases the values for alloys. The 77 K values for German silver, Manganin and Constantan alloys range from 12.5 to  $17 \text{ W}/(\text{m}\cdot\text{K})$ .

Thermal Expansion: The linear thermal expansion coefficients for unalloyed copper decrease from  $17.7 \times 10^{-6}/\text{K}$  at 294 K to  $8.4 \times 10^{-6}/\text{K}$  at 80 K. The corresponding total contraction is 0.302%. For the same temperature interval, the contractions for alloys range from 0.245% for Constantan to 0.350% for yellow brass (Cu-35Zn).

#### 4.6. Nickel and Its Alloys

Nickel and its alloys (fcc structure) offer excellent low temperature properties. Commercially pure nickel is ferromagnetic and corrosion resistant; it is a fair electrical conductor, and a crucial alloy element in cryogenic steels [96]. Nickel alloys offer high elastic moduli and medium-to-high strengths, but costs are high, and fabrication is complicated.

##### a. General Information

Designations for nickel and its alloys usually consist of generic names and three-digit numbers. Every alloy series contains precipitation hardening as well as non-heat-treatable grades. Unalloyed nickel, as well as members of the Ni-Cu, Ni-Mo, Ni-Cr, Ni-Cr-Mo, Ni-Ce-Fe, and Ni-Fe-Cr alloy system are candidates for cryogenic service. The superalloys are of greatest potential for NTF primary structural applications.

Superalloys are precipitation-hardening alloys containing chromium. Although originally developed for elevated temperature service, the superalloys also exhibit excellent cryogenic properties owing to their austenitic structure. Cryogenic temperatures lead to further strengthening, with little or no loss of ductility and toughness. Previous uses include aerospace pressure vessels at 20 K and torque tubes for superconducting machines at 4 K. Superalloy metallurgy is complex, however, and good quality control is needed in processing and fabrication.

##### b. Preferred Alloys

Commercial purity nickel (Nickel 200, 99.5%Ni) is a fair electrical conductor, finding applications in strain and temperature gages as well as liner materials for filament-wound pressure vessels. The nominal composition of Nickel 200 is 99.5Ni-0.08C-0.18Si-0.18Mn-0.2Fe-0.13Cu-0.005S, plus some cobalt. The yield stress is 144 MPa at 300 K.

The basic Ni-Cr alloy is Inconel 600 (Ni-15.5Cr-8Fe) which offers properties similar to those of annealed AISI 304 stainless steel. Inconel 625 (Ni-21.5Cr-2.5Fe-0.2Al-0.2Ti-9Mo-3.65Cb + Ta) is a modification which can be precipitation-hardened to a 300 K yield

stress of 413 MPa, or twice the value for Inconel 600. Still higher strengths are obtained using Inconel 706 (Ni-16Cr-37.3Fe-27Al-1.66Ti-2.95Cb&Ta), 718 (Ni-19Cr-18.5Fe-0.9Ti-3Mo-5.1Cb&Ta), and X-750 (Ni-15.5Cr-7Fe-0.7Al-2.5Ti-0.95Cb&Ta) superalloys.

In view of high strength and toughness, Inconel 718 and Inconel X-750 are currently the most widely used superalloys. Inconel 718 offers the highest yield stress, 1172 MPa at 300 K, whereas Inconel X-750 offers higher toughness at a slight reduction of strength. Inconel 706 is substituted for these alloys when better machinability and fabricability is desired. Several other nickel alloys have been used for cryogenic service [102]. The best known Ni-Cu alloy is Monel K-500 (66.5 Ni-28.5Cu-0.13Cu-0.75Mn-1.0Fe); it has been used in liquid oxygen lines and manifolds where complicated shapes must be formed from soft sheet, then welded, then aged to higher strength. Hastelloy B (61Ni-2.5Co-28Mo-1Cr-5Fe-0.05C) has been used in aerospace applications spanning both high and low temperature extremes. Hastelloy X (47Ni-1.5Co-9Mo-22Cr-18.5Fe-0.1C) provides better castings, compared with Inconel X750. Incoloy 903 (38Ni-15Co-3Cb-1.4Ti-0.7Al-Fe bal) is known for its relatively low expansion coefficient of  $7.2 \times 10^{-6}/K$  at room temperature.

#### c. Fabrication

Nickel base alloys, particularly the superalloys, are difficult to fabricate. The superalloys were designed to resist elevated temperature deformation, so forming temperatures are necessarily high. Casting temperatures are also high, and castings represent some sacrifice of mechanical properties compared with wrought products, although the associated heat treatments are often less complicated.

Nickel alloys are also difficult to machine, owing to high hardness and high work-hardening rates. Superalloys are best machined in the solution-annealed or hot-worked conditions. To maintain critical dimensions, the proper procedure is to machine slightly oversize, age-harden, then finish to size. This anticipates the 0.09% contraction during precipitation hardening, and better surface finish is obtained. In some cases, electrochemical, discharge, ultrasonic, electron beam, or chemical mill machining may be more economical than conventional machining.

The most common welding process for nickel alloys is gas tungsten arc, but shielded metal arc, gas metal arc and oxyacetylene flame are possible alternatives. Components are usually joined in the solution-treated condition, then postweld heat-treated. Weld processes are similar to those for stainless steels. Weld ductility and toughness are generally inferior to base metal properties, although strengths may be equivalent. Nickel alloys can also be brazed, but copper, rather than silver, brazing alloys are preferred if precipitation-hardening treatments are to follow.

Inconel 718 and Inconel X750 are heat-treated as follows: Inconel 718 is solution-treated (1256 K, air cool) and double-aged (992 K, 8 h, furnace cool; 894 K, 20 h, air cool). Inconel X-750 is solution treated (1255 K, 1 h, air cool), and double-aged (1144 K, 25 h, air cool/977 K, 20 h, air cool). Delayed hardening gives Inconel 718 better resistance to postweld strain-age cracking, compared with Inconel X750.

#### d. Availability

Superalloys are available primarily as aircraft quality sheet or forged bar. Plate is not available. Owing to high nickel content, difficult forming, machining, and heat treatments, superalloys are among the most expensive structural alloys.

#### e. Mechanical and Physical Properties

The Inconel superalloys are the best characterized Ni-base alloys. The existing fracture mechanics data base [97-101] for Inconel 718 and Inconel X-750 alloys can be readily translated to NTF applications.

Tensile: Yield stress at 300 K range from 117 MPa for annealed, commercially pure nickel to 1172 MPa for precipitation-hardened Inconel 718. Between 300 and 77 K, commercially pure nickel shows a yield stress increase of 117 to 137 MPa, a



tensile strength increase of 413 to 593 MPa, and an elongation increase of 46 to 52%. For the same temperature range, Inconel 718 shows a yield stress increase of 1172 to 1342 MPa, a tensile strength increase of 1404 to 1649 MPa, and nearly constant elongation at 20%.

Elastic: Young's modulus for unalloyed nickel is 225 GPa at 295 K. The increase at 77 K is approximately 6%. Nickel alloys exhibit slightly lower moduli, but similar temperature dependences [103-104].

Impact: Charpy impact values for nickel-base alloys are only moderately temperature sensitive, and large decreases at cryogenic temperatures are not observed.

Fatigue: Nickel alloys offer improved fatigue resistance at cryogenic temperatures, as do other fcc metals. Inconel 718 for example has a 77 K fatigue strength of 965 MPa at  $10^6$  cycles, which corresponds to 60% of the ultimate strength [27]. In strain cycling fatigue Inconel 718 shows improvement at 77 K, compared to 300 K, at all cycle lives between 100 and  $10^6$  [25]. Fatigue crack growth rates for Inconel 718 and X-750 superalloys are shown in Fig. 3.4.

Fracture Toughness: A substantial fracture mechanics data base has been built up for Inconel X-750 and Inconel 718 base metal and welds [97-101]. Fracture mechanics analysis is advisable for primary structures since superalloys may fail in a relatively brittle manner when cracks are present in thick sections. Reported  $K_{IC}$  values for Inconel X-750 and Inconel 718 fall in the range 70 to 250  $\text{MPa}\cdot\text{m}^{1/2}$  at 295 K. At 77 K there is a 7% increase of  $K_{IC}$  for Inconel 718 and a 10% decrease for Inconel X-750. Significant variability in  $K_{IC}$  can be expected owing to heat-to-treat variations [98]. Best toughness properties are obtained in heats not having grain boundary carbides.

Thermal Conductivity: The thermal conductivity of commercially pure nickel increases from about 78 W/(m·K) at 300 K to 74 W/(m·K) at 77 K. Inconels, Hastelloys, and Monels show values in the ranges 0.1 to 0.23 at 300 K and 0.058 to 0.16 at 77 K.

Thermal Expansion: Between 293 and 80 K, commercially pure nickel contracts 0.21%, which is near values for ferritic steels. The contractions for many nickel alloys are within  $\pm$  of pure nickel [38].

#### 4.7 Titanium and Its Alloys

Unalloyed titanium and extra-low-interstitial (ELI) Ti-5Al-1.5Sn and Ti-6Al-4V alloys are qualified for cryogenic applications where higher material and fabrication costs are justified by the physical and mechanical properties obtained. Advantages are excellent corrosion resistance, low thermal conductivity, low density (about 4/7 that of stainless steel), and high cryogenic strength. On the other hand, titanium components are relatively difficult to fabricate, and the medium strengths at room and elevated temperatures are disadvantageous for some applications. The elastic modulus of titanium is higher than that of aluminum alloys, but lower than that of steels.

##### a. General Information

Titanium alloys have alpha (hcp), beta (bcc), or alpha + beta crystal structures. Alloys in which the hcp phase predominates are useful at cryogenic temperatures, but all beta alloys are brittle at low temperatures and worthless for applications at 77 K. Since the mechanical behavior is sensitive to purity, the Ti-5Al-2.5Sn and Ti-6Al-4V alloys must be obtained as ELI grades. Note that normal grade Ti-5Al-2.5Sn and Ti-6Al-4V alloys are obsolete for cryogenic service because their higher impurity levels compromise cryogenic ductility and toughness. Since cracked ELI titanium alloy components are apt to fail in a brittle manner at cryogenic temperatures a fracture mechanics analysis may be necessary to verify structure integrity.

## b. Preferred Alloys

Unalloyed titanium is available in three grades and is used in the annealed condition. The 295 K yield stress of the three grades are nominally 379 MPa, 488 MPa, and 517 MPa. Selection of one of these three grades of unalloyed titanium should be based on the knowledge that strength increases while ductility decreases with decreasing purity. NTF applications for unalloyed titanium would be those for which strength is not the primary consideration, as in the construction of aerodynamic surfaces. Unalloyed titanium is less expensive, lower in strength, and about twice as thermally conductive as the Ti-5Al-2.5Sn(ELI) and Ti-6Al-4V(ELI) alloys. Specifications for titanium alloys are listed in Table 4.5.

Ti-5Al-2.5Sn(ELI) and Ti-6Al-4V(ELI) alloys are used where high strength-to-thermal conductivity or strength-to-weight ratios are required. Ti-5Al-2.5Sn(ELI) has a single-phase hcp structure and is used in the annealed condition. The 295 K yield stress of Ti-5Al-2.5Sn(ELI) is 750 MPa. Past applications specified furnace cooling from the annealing temperature of 1088 K, but air cooling apparently would confer superior fracture toughness [105].

Ti-6Al-4V(ELI) has a two-phase microstructure consisting predominantly of hcp and some bcc. This alloy can be aged, but is typically used in the annealed condition to maximize toughness for cryogenic service. A recrystallization anneal at 1200 K followed by furnace cooling results in a 295 K yield stress of 840 MPa. Past uses for Ti-5Al-2.5Sn(ELI) and Ti-6Al-4V (ELI) include large welded cryogenic pressure vessels for aerospace propellant systems, as well as small laboratory cryostat components. For applications at 77 K or above, Ti-6Al-4V (ELI) is more commonly selected because it offers adequate toughness and higher strength than Ti-5Al-2.5Sn(ELI).

## c. Fabrication

Titanium alloys are more difficult to machine than most steels, except austenitic stainless steels. Difficulty in processing arises owing to contamination by atmospheric impurities. For this reason electron beam welding in vacuum is preferable to gas-metal-arc or gas tungsten-arc welding. Electron beam welding is more expensive, but it reduces the risk of contamination and yields more ductile welds.

## d. Availability

Unalloyed titanium and Ti-6Al-4V(ELI) are readily available in standard product forms. Billets of Ti-5Al-2.5Sn(ELI) are also available in sections 10 cm square or 10 cm in diameter, but smaller forms may be more difficult to obtain. Prices for ELI grade alloys range from about \$2.7/kg for plate or billet to about \$4.5/kg for sheet. Unalloyed titanium may be several dollars per pound cheaper.

## e. Mechanical and Physical Properties

The mechanical properties of titanium are strongly temperature dependent and sensitive to impurities (O, H, N, C, and Fe). Accurate fracture mechanics data are needed in critical applications.

**Tensile:** The 295 K yield stresses for the recommended alloys range from 379 MPa for unalloyed titanium to 140 MPa for Ti-6Al-4V(ELI). Temperature reductions to 77 K approximately double the yield stress, while ductility is moderately reduced.

**Elastic:** Young's modulus for unalloyed titanium at 295 K is 114 GPa, slightly lower than that of copper. The 295 K Young's moduli for Ti-6Al-4V and Ti-5Al-2.5Sn alloys are nearly equivalent at 111 GPa. Temperature reductions to 77 K increase the moduli by 9 or 10% [106].

Table 4.5. Titanium alloy specifications.

Material	Product form	Applicable specifications		
		Federal	ASTM	AMS
Commercial Purity Titanium				
A 40, 55A	Sheet, strip, plate	MIL-T-9046, I, A	ASTM B 265-72	4902B
A 55, 65A	Sheet, strip, plate	MIL-T-9046, I, C	ASTM B 265-72	4900F
A 70, 75A	Sheet, strip, plate	MIL-T-9046, I, B	ASTM B 265-72	4901G
Ti-6Al-4V, ELI	Sheet, strip, plate	MIL-T-9046, III, D	ASTM B 265-72	4907C
	Bar, forgings, rings	MIL-T-9047, Comp. 7	ASTM B 381-69	4930B
Ti-5Al-2.5Sn, ELI	Sheet, strip plate	NA	ASTM B 265-72	4909C
	Bar, forgings, rings	MIL-T-9047, Comp. 3	ASTM B 381-69	4924C



Impact: Charpy impact energies for titanium alloys decrease at cryogenic temperatures. A typical CVN value for annealed Ti-6Al-4V(ELI) is 19.5 J at 77 K.

Fatigue: Within the data scatter of replicate tests, fatigue crack growth rates for Ti-5Al-2.5Sn and Ti-6Al-4V alloys are temperature insensitive between 295 and 76 K providing the stress intensity factors remain at intermediate values, not approaching  $K_{IC}$ . Stress controlled S-versus-N fatigue data show improvement as temperature is reduced between 295 and 77 K.

Fracture Toughness: Flawed titanium alloys are brittle at 77 K, even though the same alloys are ductile in the unflawed or unnotched condition. The critical flaw sizes at 77 K are small. The  $K_{IC}$  values for titanium alloys decrease with decreasing temperatures [107]. Typical  $K_{IC}$  values for recrystallization annealed Ti-6Al-4V(ELI) are  $100 \text{ MPa}\cdot\text{m}^{\frac{1}{2}}$  at 295 K and  $61 \text{ MPa}\cdot\text{m}^{\frac{1}{2}}$  at 77 K [108]. Typical values for annealed, air-cooled Ti-5Al-2.5Sn(ELI) alloy are  $101 \text{ MPa}\cdot\text{m}^{\frac{1}{2}}$  at 295 K and  $75 \text{ MPa}\cdot\text{m}^{\frac{1}{2}}$  at 77 K.

Thermal Conductivity: Thermal conductivities of Ti-6Al-4V and Ti-5Al-2.5Sn at 295 K are about half the values of unalloyed titanium, stainless steel, and 9%Ni steel. The temperature dependence of thermal conductivity for Ti-5Al-2.5Sn (normal grade) shows a decrease from  $8.17 \text{ W}/(\text{m}\cdot\text{K})$  at 300 K to  $4.4 \text{ W}/(\text{m}\cdot\text{K})$  at 80 K.

Thermal Expansion: The linear thermal expansion coefficients for titanium and the recommended alloys at 300 K are  $8.4$  to  $9.4 \times 10^{-3}/\text{K}$ , which is nearly equivalent to values for 9%Ni steel. Total contraction between 295 and 90 K is 0.14 to 0.18%.

#### 4.8. Cobalt and Magnesium

Cobalt superalloys (hcp structure) have served in some cryogenic aerospace applications but are not recommended for NTF applications in view of high cost, limited availability, poor fabricability, lack of design data, and lack of documented service experience.

Magnesium and its alloys were once considered attractive for high strength-to-density ratios, but Charpy V-notch impact energies for all cast and wrought Mg metals are too low (1.35 to 9.5 J) for service at temperatures between 295 and 77 K [109-110].

## 5. METALS FOR BRAZING AND SOLDERING

Most metals, including stainless steels copper, nickel, aluminum, and titanium alloys, can be brazed or soldered under appropriate conditions. The purpose may be to establish electrical, mechanical, or thermal contacts, or simply to produce a seal.

Brazed and soldered joints are formed by fusing a thin layer of nonferrous filler metal into a narrow gap between two base metal components. Bonding results from the wetting and dissolution of the base metal by the filler, without fusion of the base metal. The only distinction between brazing and soldering is the temperature required. By convention, joining at temperatures greater than 725 K is brazing, while joining at lower temperatures is soldering [111-112].

### 5.1. Fillers and Fluxes

For both brazing and soldering, fillers having an eutectic composition are favored because they melt at relatively low temperatures. Care must be taken if noneutectic compositions are used because noneutectics melt over a range of temperatures during which the filler exists as a two-phase solid/liquid "paste." Pasty fillers do not flow readily into narrow gaps, and leaks or voids owing to lack of penetration may result. Specific fillers are recommended in the following sections.

The filler must wet the base metals to be joined. Wetting depends on the affinities of specific materials and is promoted by using fluxes to remove surface oxide films and protect the heated metals from the atmosphere. Borax or boric acid fluxes are generally used for brazing. There are three alternatives for soldering: 1) hydrochloric or phosphoric acid fluxes, 2) pastes of petroleum jelly, and 3) rosins or resins. Manufacturer's recommendations should be followed in selecting fillers and fluxes for specific applications.

### 5.2. Brazing

The advantages and disadvantages of welding, brazing, and soldering are listed in Table 5.1. Brazing is preferable to welding where dissimilar metals must be joined conveniently and rapidly, without fusing the base metals. Brazed joints are stronger, more permanent, and more resistant to thermal cycling than are soldered joints. On the other hand, the permanency of a brazed joint can be disadvantageous, and care must be taken that the brazing temperatures (875 to 1150 K) do not damage the base metals. For example, brazing may anneal copper or aluminum alloys, sensitize austenitic stainless steels, or volatilize the lead in leaded brass. Consequently, it may be impossible to replace a brazed joint without damaging a complex apparatus [113-115].

Brazing is also referred to as hard soldering or silver soldering because the recommended brazing fillers are usually silver alloys which are stronger than solders. For most cryogenic applications, brazing alloys with 45 to 72% silver are superior to brazing alloys containing little or no silver [116]. Silver brazing alloys are available in the form of sheet, wire, powder, and preforms. Copper, nickel, and gold brazing fillers are also available for special purposes.

The most frequently used commercial silver brazing alloys for cryogenic service are the 50%Ag-15.5%Cu-16.5%Zn-18%Cd and 45%Ag-15%Cu-16%Zn-24%Cd alloys, which are included in AWS A5.8-76 specifications. The cadmium additions create alloys of near-eutectic composition, with melting points in the range 893 to 908 K. These alloys have low viscosity and flow readily into joints with gaps from 0.025 to 0.127 mm. Seligmann and Sarwinski [117] report a 0.433% thermal contraction for one such alloy between 295 and 4 K. Thermal conductivity is about 60 W/(m·K) at 200 K, and 33 W/(m·K) at 77 K [118]. Electrical resistivity at 295 K is about 6 to 10  $\mu\Omega\text{cm}$ , and electrical conductivity is about 18 to 28% IACS. The 295 K thermal expansion coefficients of about  $1.8 \times 10^{-5}/\text{K}$  closely match the coefficients of coppers, brasses, bronzes, and AISI 304 stainless steel.

The tensile properties of the Ag-Cu-Zn-Cd fillers are unreported, but the tensile strengths of Ag-Cu-Zn brazing alloys at 300 K range from 234 to 417 MPa. Reduction in

Table 5.1. Comparison of welding, brazing, and soldering.

	Welding	Brazing	Soldering
Advantages	High strength High thermal cycling resistance Few corrosion problems Large sections	Joins dissimilar metals Little or no warping Thin sections Good appearance Stronger than soldered joints	Joins dissimilar metals Little or no warping Thin sections Good appearance Low heat input Inexpensive Easy to disassemble
Disadvantages	Base metal is melted High heat input Distortion Expensive	More expensive than soldering Difficult to disassemble Higher heat input than soldering	Low thermal cycling resistance Corrosive fluxes Low strengths

area is typically 15 to 50%. Good mechanical properties are retained at cryogenic temperatures, since silver and copper have fcc structures. Some available data at temperatures from 295 to 77 K show 20 to 60% improvements in tensile strength of Ag-Cu-Zn brazing alloys and only minor effects on ductility [119-122].

### 5.3. Soldering

Compared to brazing alloys, the melting points of solders are relatively low. Therefore, soldered joints are easily formed, disassembled, and reformed without thermal damage to neighboring apparatus. Solders are also less expensive than brazing alloys. On the otherhand, soldered joints are weaker than brazed joints, less resistant to thermal cycling, and limited in use at elevated temperatures near the melting point. Aluminum, magnesium, and chromium are difficult to solder, except by means of special techniques and fluxes. Copper alloys are easiest to solder.

The most common solders are Pb-Sn alloys which melt at temperatures ranging from 456 K (the 37%Pb-63%Sn eutectic) to 599 K (unalloyed Pb). Other solders contain lead, tin, indium, bismuth, cadmium, mercury, or gallium. Most of these elements are volatile and toxic, especially Cd, Hg, and Pb. Therefore, care must be taken during melting, and overheating should be avoided.

Tensile, shear, and impact data for solders are reported at temperatures between 20 and 373 K [120, 123-128]. In general, the mechanical properties of solders are sensitive to composition and testing rate. For example, Thwaites and Hampshire [128] show two- or three-fold increases of tensile strength for Pb-Sn solders at 295 K as the testing rate is varied from 0.0083 to 0.83 mm/s. Rate effects are also sizable at cryogenic temperatures [123].

Lead-tin Solders. Selected mechanical property data for Pb-Sn solders are shown in Figs. 5.1 and 5.2. All Pb-Sn solders are ductile at room temperature, but tin-rich solders exhibit low temperature ductile-to-brittle transitions. The tin-rich solders offer higher strength and creep resistance for room or elevated temperature service, but their poor low temperature ductility makes them undesirable for cryogenic service, except in applications where mechanical properties are unimportant [125].

Lead additions increase the amount of fcc phase in solders, while decreasing the ductile-to-brittle transition temperature. For example, unalloyed tin (tetragonal structure) is brittle at 163 K, whereas 50%Pb-50%Sn solder is brittle at 123 K. Ainsworth [129] concludes that a lead content of 80% or more is required to retain ductility and toughness in Pb-Sn solders at 77 K. Nevertheless, 40%Pb-60%Sn solder is frequently used at 77 K if mechanical performance is not critical.

The strength, ductility, and impact properties of unalloyed lead (fcc structure) continuously increase between 295 and 20 K, but unalloyed lead is a poor solder because it does not wet many metals. Some tin is needed to promote wetting and compatibility with rosin fluxes. Consequently, the optimum ductile solders for extreme cryogenic service are alloys such as 90%Pb-10%Sn, 97.5%Pb-1%Sn-1.5%Ag, and 93.5%Pb-5%Sn-1.5%Ag. These solders show improved strength and high ductility at temperatures down to 77 K [126].

Although some thermal property measurements for 40 to 60% Pb solders are reported [16, 117, 130], such data are rare, and the cryogenic thermal properties for most solders must be estimated from the pure metals.

Bismuth Solders. Solders containing 50% Bi or more expand on solidification, whereas most metals and alloys contract. Therefore, special uses in sealing applications have been found for bismuth solders, despite the fact that they are weak and brittle at room and cryogenic temperatures.

Wood's metal (50%Bi-25%Pb-12.5%Sn-12.5%Cd) is a bismuth solder noted for its exceptionally low melting point of about 335 to 342 K. Thus, Wood's metal can be used to form joints without disturbing neighboring Pb-Sn soldered joints. However, Wood's metal is



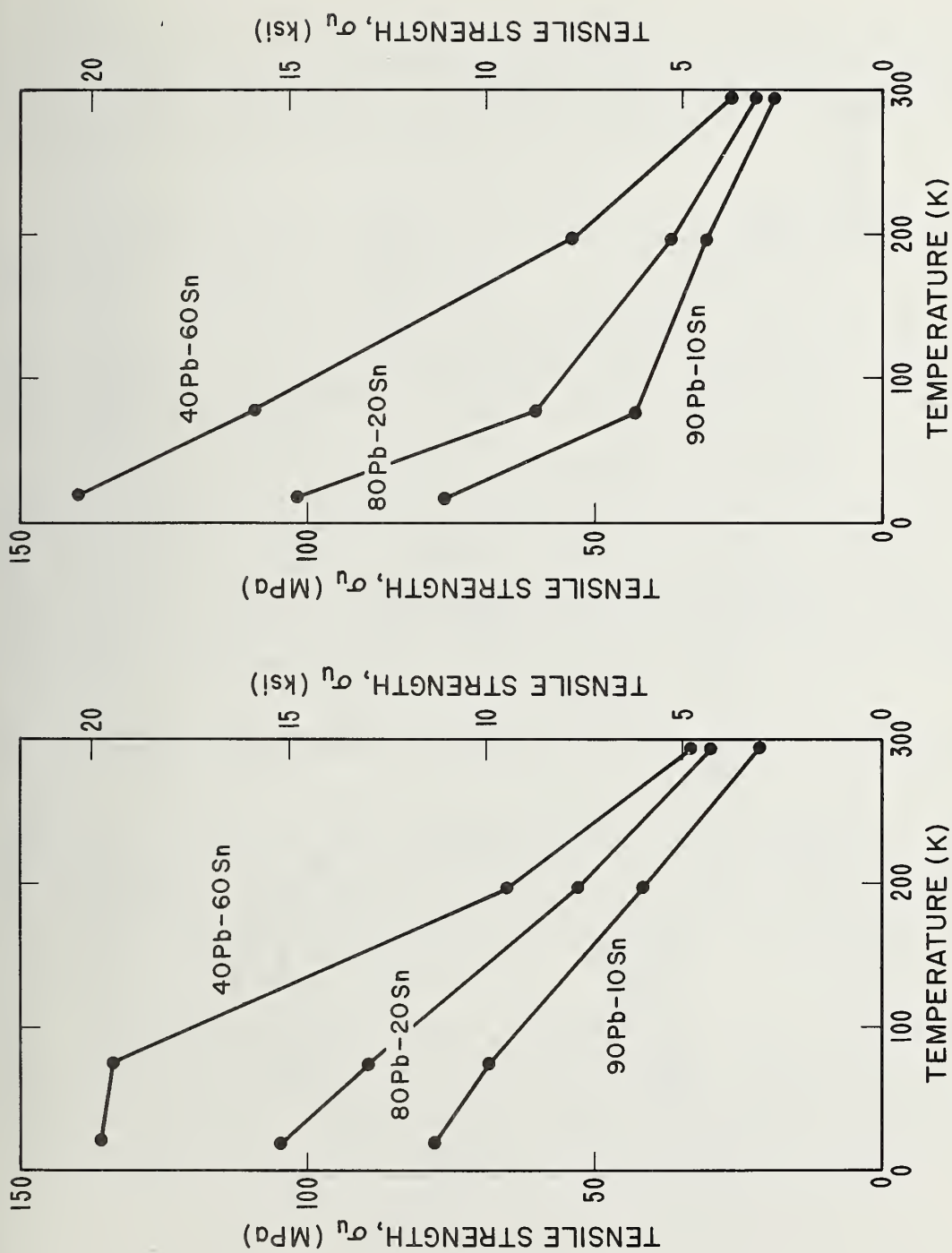


Figure 5.1. Cryogenic strengths of lead-tin solders.



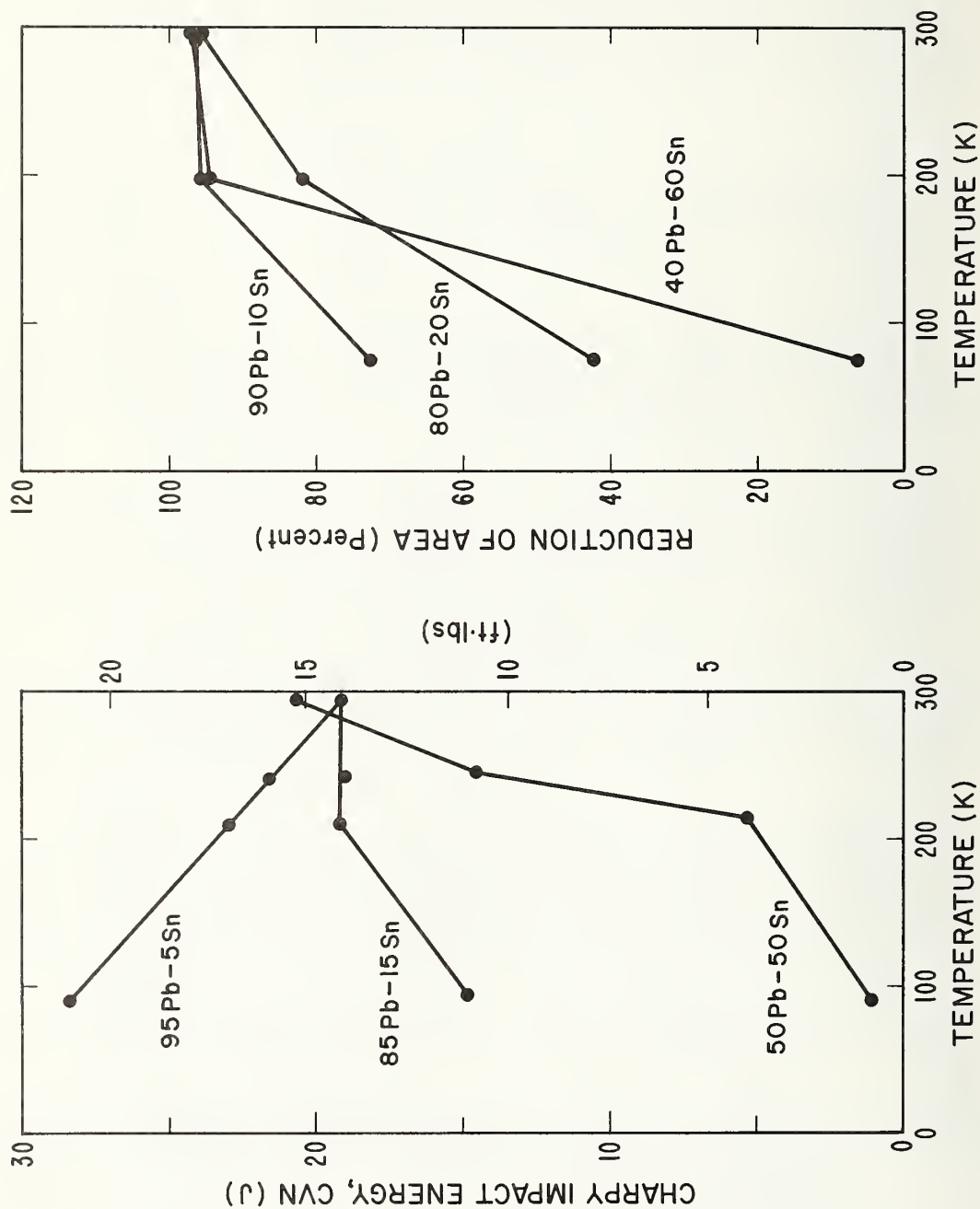


Figure 5.2. Cryogenic effects on the ductility of lead-tin solders.

only about half as strong as Pb-Sn solders, and it has a low resistance to thermal cycling. This solder also has a melting point lower than the 367 K temperature encountered in the NTF. Seligmann and Sarwinski [117] report a 0.582% thermal contraction for Wood's metal between 295 and 4 K.

Indium Solders. The most outstanding characteristic of indium is its ability to wet diverse materials. Thus, indium solders can be used for a variety of glass-to-glass, glass-to-ceramic, and ceramic-to-metal joints. Indium solders acceptable for cryogenic service are 100%In, 90%In-10%Ag, and 50%In-50%Pb. These solders are noted for melting points ranging from 416 to 573 K, low strength, excellent ductility at all temperatures, and higher thermal conductivity compared to Pb-Sn solders.

#### 5.4. Joints

A brazed or soldered joint consists of two base metal components, a narrow diffusion zone, and a filler zone. Lap or butt joints are common in service, but ring-and-plug joints are recommended for research [128].

The mechanical properties of brazed or soldered joints cannot be predicted simply from the properties of the fillers themselves. Joint properties are a function of base metal properties, joint geometry, and type of stress applied. Since the filler is constrained by base metal components, the joint may sustain higher stress than expected for fillers in bulk form [131].

Temperature cycling frequently causes failure in soldered joints. To minimize this problem, the solder and base metal thermal expansion coefficients should be matched as closely as possible.

The 300 K shear strengths of soldered joints typically range from 10 to 50 MPa and are less than or equal to the bulk solder tensile strength. Raising the temperature from 295 to 373 K may decrease the shear strengths of soldered joints by 25 to 80% [128]. Brazed joints give higher strengths, with butt tensile strengths at 295 K ranging as high as 1100 MPa. Representative shear strengths for brazed copper joints at 77 K range from 123 to 207 MPa, which is equal to or greater than the strengths at 295 K.

As a guide to the mechanical behavior of joints at cryogenic temperatures two experimental studies are noted:

1. Jaffee, Minarcik, and Gonser [124] tested copper tubing joints made with Pb-Sn solders. The tubes (25.4 mm ID, 1.6 mm wall) were soldered using standard sleeve fittings, then tested at 300, 244, and 91 K. Regardless of solder type, the joint-breaking loads increased linearly with decreasing test temperature. For high-tin solders (75%Pb-25%Sn and 50Pb%-50%Sn), the low temperature fractures occurred in the joint. For high-lead solders (97.5%Pb-2.5%Sn and 95%Pb-5%Sn), fractures tended to occur in the tubes or fittings. But the breaking loads at a given test temperature were nearly equivalent to and virtually independent of the solder used.

2. Liebenberg [132] tested single-overlap joints made from 6.25 x 37.5 x 76.2 mm brass plates and a 60%Sn-40%Pb solder. The ultimate shear strength at 295 K immediately after soldering was 34 MPa (5 ksi). However, room temperature aging for 50 days led to a 25% reduction of shear strength. Ten thermal cycles between 295 and 77 K decreased the joint shear strengths by an additional 20%. The shear strengths at 295 and 20 K were nearly equivalent. This apparent temperature independence of shear strength is also reported for silver-brazed copper joints tested between 295 and 77 K [133].

## 6. METALS FOR PLATING AND PROTECTION

Plating serves functional and aesthetic purposes. Some common cryogenic steels, such as Fe-9Ni steel, will rust after cryogenic testing unless plated or dried in an air jet to quickly remove condensed moisture. Plating can also be used to improve surface condition and appearance, prevent galling between nuts and bolts, or embed lead wires and instrumentation.

The satisfactory cryogenic plating metals listed in Table 6.1 have fcc structures. Non-fcc metals, such as tin, are brittle at cryogenic temperatures and are, therefore, avoided. The platings in Table 6.1 have served in aerospace applications at temperatures to 77 K or lower [134]. In selecting a plating, materials compatibility and cost should be considered. To avoid corrosion, the plating and substrate should be ranked appropriately on the electromotive series. Gold, platinum, rhodium, and aluminum platings are expensive. Nickel and silver are more commonly used.

Table 6.1. Platings for cryogenic service.

Plating	Hardness at 295 K (Rockwell B or C, as noted)
Aluminum	<56R <sub>B</sub>
Copper	<97R <sub>B</sub>
Gold	26 to 77 R <sub>B</sub>
Indium	NA (very soft)
Lead	NA (very soft)
Nickel	76 to 93 R <sub>B</sub>
Platinum	NA
Rhodium	43 to 72 RC
Silver	<88 R <sub>B</sub>

## 7. NONMETALS

Wood, glass, ceramics, and polymers are discussed in this section. These materials find special applications in wind tunnel models, but are less commonly used than metals.

### 7.1. Wood

Wood is a porous natural composite of cellulose and lignin. Wood is rarely used at cryogenic temperatures, but it serves as a structural insulator in special applications. Most thermal insulators are heavy, except wood and foamed plastics. Foamed plastics, however, have low strength. Therefore wood may be the best choice where a balance of strength and thermal insulation is required.

Current applications for wood include cryogenic storage tank and pipeline supports. In some designs balsa blocks are used to support the entire weight of liquefied natural gas storage tanks [135]. Plywood facings and balsa frameworks are used in combination with foamed plastics in modern thermal insulation and support systems for LNG tankage. Wood frameworks overlaid with fiberglass are used for honeycomb structures. In NTF applications, wood can be used similarly, if it is sealed against moisture and if sizable thermal contraction can be tolerated.

#### a. General Information

The mechanical and thermal properties of wood are proportional to density and strongly dependent on moisture content and orientation. Orientation is specified with respect to the three orthogonal axes: longitudinal (parallel to grain), radial, and tangential. For example, the linear thermal expansion coefficient for Douglas fir at room temperature is  $2.7 \times 10^{-6}/K$  in the longitudinal direction compared with  $27 \times 10^{-6}/K$  in the tangential or radial directions [136]. Similarly, longitudinal strength may be ten times greater than radial or tangential strength. The anisotropy can be reduced using plywood.

Moisture in wood significantly reduces room temperature strength. Moisture also causes swelling and warping, and general loss of dimensional tolerances. Moreover, premature failure at low temperatures may result from high water content [136, 137]. This is a problem since cryogenic apparatus will condense moisture from ambient, humidified air.

#### b. Specific grades

Among the hundreds of species of soft and hardwoods, balsa is unique. The lightest of woods, balsa is commercially available in densities ranging from 88 to 192 kg/m<sup>3</sup>. End-grained balsa (the strongest orientation) can support 6.9 MPa while providing lower thermal conductivity than many other woods, and lower thermal expansion than insulating plastics. Some cryogenic mechanical properties of balsa are reported by Schwartzberg [138].

Plywoods are laminates of an odd number of wood plies glued together at angles of up to 90°. Compared with natural wood, plywoods offer several advantages, such as higher resistance to splitting, reduced anisotropy, and greater dimensional stability in the presence of moisture.

Impregnated wood is a unique material, well suited for cryogenic environments. In the impregnation process, wood is densified by heating and pressing at high pressures, then polymerized by irradiation. Impregnation with methyl methacrylate, styrene, ethylacrylate, or phenolic resin improves strength while obviating the moisture problem. Impregnated wood can be placed in direct contact with water without losing dimensional stability, since moisture absorption is less than 1%. After impregnation, tests at room temperature show improved stiffness (34%), compressive strength (600%), tensile strength (18-86%), and hardness (350 to 1000%). Toughness usually decreases, however, because irradiation damages some woods, particularly hardwoods. Impregnated wood has been used in bearing insulating block applications at compressive design stresses up to 83 MPa and at shear stresses up to 6.9 MPa. The primary disadvantage is higher cost [139, 140].



### c. Mechanical and Physical Properties

The mechanical and physical properties of wood at cryogenic temperatures are poorly documented, but data at 77 K for balsa, spruce, fir, beech, beech plywood, and impregnated beech are reported [136, 137, 139, 140]. Representative data trends are illustrated in Figs. 7.1 and 7.2. Dried natural woods generally show improved mechanical properties at cryogenic temperatures. The compressive strengths for longitudinal orientations at 77 K range from 28 MPa for balsa to 158 MPa for beech. By comparison, the compressive strengths for impregnated and densified beech veneers at 77 K may reach 321 MPa. Adler's results for impregnated wood between 293 and 81 K indicate approximately a 20% reduction in tensile strength, shear strength, and impact values [139].

Limited thermal conductivity data for some woods and wood fiber products at selected temperatures between 367 and 77 K are tabulated by Loser, Moeller, and Thompson [141]. A gradual decrease in thermal conductivity is expected at low temperatures. The thermal conductivity values for balsa wood decrease from 0.04 to 0.026 W/(m·K) between 201 and 90 K. The thermal conductivities of an impregnated and densified wood are reported to range from 2.6 to 4.5 W/(m·K) at 90 K [140]. The average thermal expansion coefficients for impregnated wood between 77 and 290 K range from  $2.5$  to  $3.5 \times 10^{-6}/\text{K}$  [139].

### d. Fabrication

Wood is readily available and easily machined using carpentry or metal-working tools. At room temperature, glued joints may be stronger than the wood itself. At cryogenic temperatures, joints may be weaker, owing to mechanical constraint or embrittlement of the adhesive.

Impregnated wood can be obtained at higher cost in sections up to 102 mm (4-in) thick. Greater thicknesses are achieved by bonding separate sections. Carbide-tipped tools are recommended in machining the very hard impregnated woods. Tolerances of  $\pm 0.381$  mm can be met.

## 7.2. Glass

Glasses are ceramics manufactured by cooling from the melt at rates too rapid to permit crystallization. Consequently a vitreous (amorphous) structure is produced, intermediate between the crystalline solid and liquid states. Silica ( $\text{SiO}_2$ ) is the major component in commercial glasses, and silica tetrahedra are the basic building blocks of glass structures, which consist of three-dimensional networks lacking long-range order.

### a. General Information

Glasses offer high heat resistance, thermal stability, insulation and transparency. Cryogenic applications include windows for spacecraft and bubble chambers, vacuum dewars for cryostats, compressive load-bearing insulators, and reinforcing fibers for composites. Brittleness is the chief disadvantage of glass. Therefore, applications involving high tensile stresses or impact loading are avoided. Glasses should be used in the annealed condition to relieve residual stresses, unless toughening treatments are applied.

Many scientific studies have been performed on glass. Fracture stresses are always lower in tension than compression, widely scattered, and highly sensitive to flaws or stress concentrators. Other undesirable aspects of mechanical behavior include the phenomenon of static fatigue and the poor thermal shock resistance of most grades.

### b. Specific Materials

Some common glasses and their compositions are listed in Table 7.1. Many compositions are unacceptable for cryogenic service, owing to poor thermal shock resistance.

E and S glass fibers are recommended for composite reinforcement applications, as discussed in section 8.3. For bulk cryogenic applications, Pyrex is favored because it has high chemical and thermal shock resistances. Typical applications for Pyrex are



Table 7.1. Glass compositions.

Material	Composition (wt%)						
	SiO <sub>2</sub>	B <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO
Fused silica	99.9	-	-	-	-	-	-
Vycor	96	3	<0.2	-	0.4	<.2	-
Pyrex	80-81	12-13	4	-	2	0.4	-
Soda-lime	60-75	-	12-20	8-20	1	-	3
E glass fibers	54	8.3	<1.2	17.5	14.5	<1.2	4.5
S glass fibers	64-66	-	-	-	24-26	-	9-11

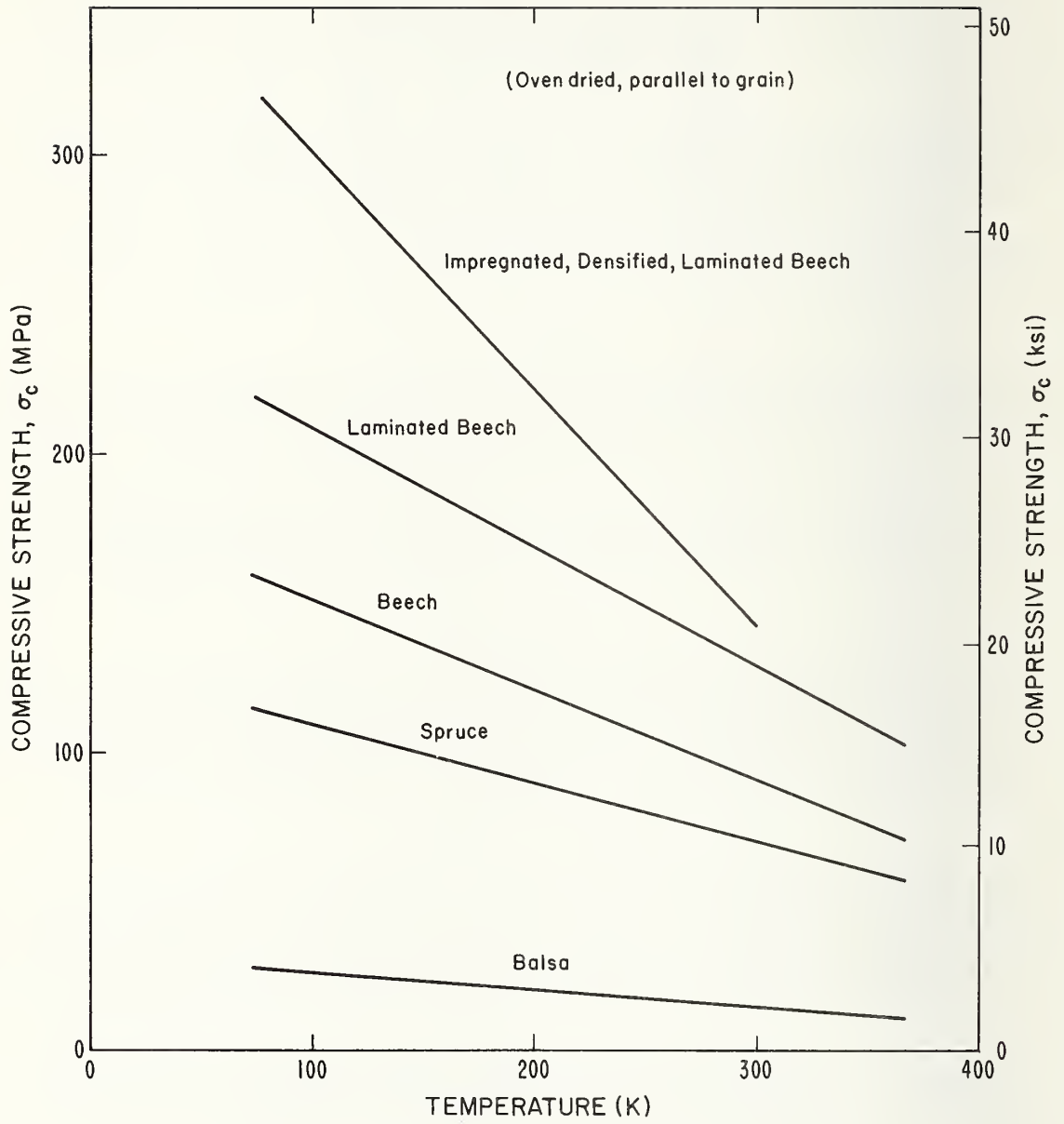


Figure 7.1. Cryogenic effects on the compressive strength of woods.

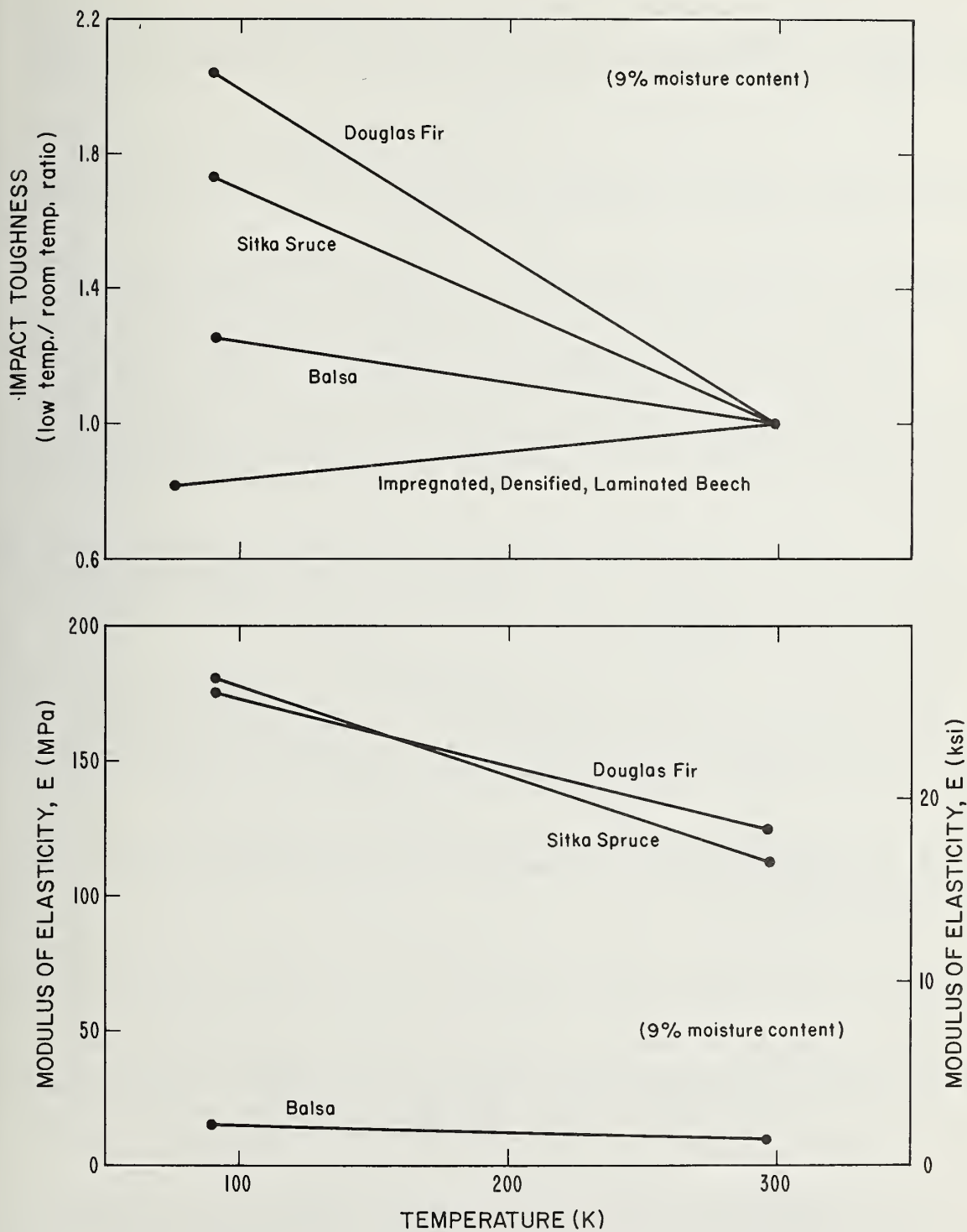


Figure 7.2. Cryogenic effects on the impact toughness and Young's modulus of woods.

dewars and windows. As a window material, Pyrex competes with clear plastics [142]. The clear plastics offer superior formability and toughness, and are lightweight, but Pyrex has superior mar resistance and dimensional stability. Impact values at 77 K are typically 0.3 J for Pyrex and 4 J for Plexiglas.

Vycor and fused silica offer high thermal-shock resistances, but they cost more than Pyrex. Fused silica glasses are used where minimum thermal expansion is required, as in dilatometer rods. Ultralow-expansion fused silica (ULE 7971) is a unique glass with the lowest thermal expansion of all.

### c. Mechanical and Physical Properties

The physical and mechanical properties of glasses correlate well with silica content. Strength, thermal conductivity, and electrical resistance tend to increase, while thermal expansion tends to decrease with higher silica content.

Strength: The strength of glass depends on specimen type, surface condition, chemical composition, thermal history, and loading rate as well as the amount of water dissolved in the bulk, absorbed on the surface, and present in the surrounding atmosphere. Hillig [143] showed that undamaged fused silica filaments may exhibit fracture stresses of up to 4.7 GPa at room temperature, but much lower values are expected for commercial, bulk glass. In bulk glass, low tensile strengths from 34.5 to 207 MPa commonly result from surface defects. This is supported by Ritter and Cooper [144], who demonstrate a sixfold increase in strength (from 413 to 2480 MPa) after chemical etching to reduce surface flaws. Therefore, high tensile strengths are theoretically possible for small, carefully prepared laboratory specimens, but low strengths are realistic for engineering purposes since commercial glasses always contain microscopic damage introduced during handling or manufacture.

Figure 7.3 depicts the temperature dependence of glass tensile strengths in the range between 76 and 400 K. Conditions varying from carefully prepared (flame-drawn, chemically polished fibers) to intentionally abraded (sandblasted) bulk glass are represented [145-148]. The fracture strengths vary widely in magnitude, but in each case the strength approximately doubles between 295 and 76 K.

Static fatigue: Glass weakens in the presence of moisture, especially at slow loading rates. This results from the hydrolysis of flaw-tip Si-O bonds, which promotes the gradual growth of existing flaws under tensile stress. At higher temperatures, the "static fatigue" process is accelerated and the strength reduction is quite dramatic: dry strengths are about double the wet strengths at 300 K [149]. Static fatigue occurs at temperatures down to 194 K, but there is little or no effect at 76 K [147].

Elastic modulus: A typical engineering value for Young's modulus of glass at 300 K is 69 GPa ( $10^7$  psi). With increasing temperature between 77 and 367 K, the moduli for Pyrex and fused silica glasses actually increase a few percent, following a linear temperature dependence [150, 151].

Electrical resistivity: Hirayama and Berg [42] report electrical resistivity data for selected glasses at temperatures between 245 and 473 K. Resistivities range between  $10^{10}$  and  $10^{18}$  ohm-cm, with the highest values for Vycor, then Pyrex, then soda-lime glass.

Thermal expansion: The thermal expansion coefficients of glasses at 295 K vary by over two orders of magnitude, from 0.2 to  $90 \times 10^{-7}/K$  [42, 152-156]. Total contraction for Pyrex between 295 and 76 K is 0.05%. In most cases the expansion coefficients decrease monotonically at temperatures down to 76 K, but fused silica glasses undergo a reversal and begin to expand at temperatures below about 200 K. Thus, ultra-low-expansion fused silica (ULE 7971), exhibits a negative expansion coefficient of  $-10 \times 10^{-7}/K$  at 76 K.

Thermal conductivity: Thermal conductivity values for most glasses in the cryogenic range are readily available [157-160]. Ratcliffe's data [158] for 22 glasses defines a band of thermal conductivity values ranging from 37 to  $86 \times 10^3$  W/(m·K) at 123 K to 61 to 146 W/(m·K) at 373 K. Ratcliffe also presents formulas for predicting values for untested

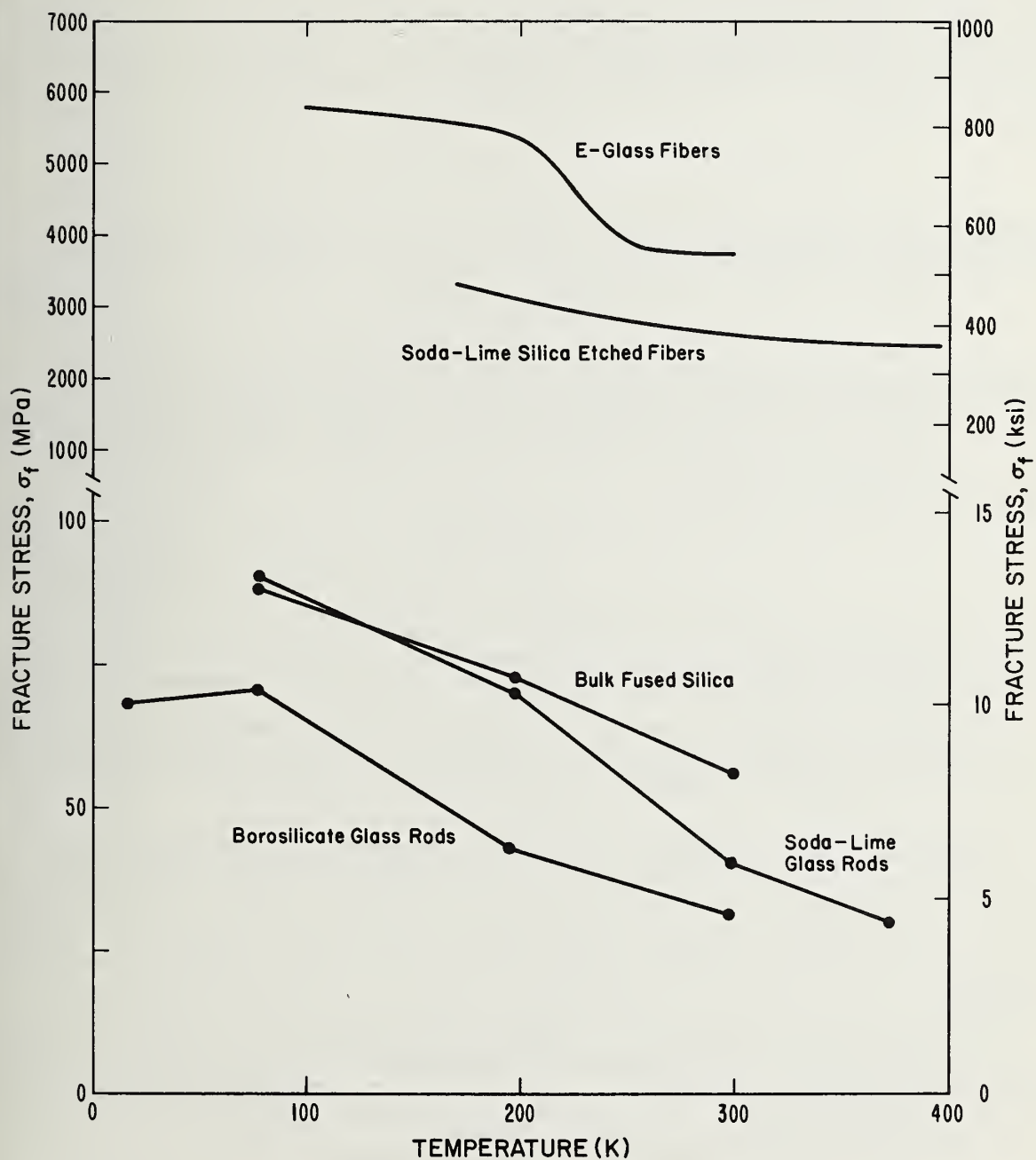


Figure 7.3. Cryogenic effects on the fracture strength of glasses.



glasses at 373, 273, or 173 K to an accuracy of  $\pm 15\%$ . Measurements at 76 K or lower are tabulated by several authors [157-160].

**Design:** If glass must be subjected to tensile or bending stresses, every reasonable attempt should be made to design conservatively. Horton's [146] approach to the design of pressurized, fused silica windows for space vehicles is exemplary. In that study, numerous flexural tests spanning the elevated-to-cryogenic temperature service range were statistically evaluated to arrive at design allowables. The test specimens were first sandblasted to assure conservative baseline data. The design allowable flexural stress obtained was 29 MPa for temperatures between 295 and 76 K. This compares to the average flexural strength measurements of 89 MPa and 56 MPa at 76 and 295 K, respectively. Tensile allowables were taken as 1/2 the value of flexural allowables, which is common procedure for ceramics.

### 7.3. Ceramics

Ceramics are inorganic materials known primarily for elevated temperature service. Cryogenic uses are rare, and only miscellaneous uses are anticipated in NTF testing. Possible applications would be insulators, bearing materials, or fillers for polymers.

#### a. General Information

Ceramics are typically brittle, weak in tension, fatigue-prone, hard, abrasive, and difficult to machine. Scatter in mechanical properties is high. On the other hand, ceramics are relatively inexpensive, strong in compression, corrosion resistant, wear resistant, electrically insulating, moderately dense, and stiff. Ceramics may be classified as: (1) glasses (2) glass-free ceramics, (3) polycrystalline aggregates in a glassy matrix, and (4) composites such as cermet. Glasses are discussed in section 7.2.; ceramic fibers have no cryogenic uses.

#### b. Specific Materials

Glass-free ceramics include mica, alumina ( $\text{Al}_2\text{O}_3$ ), magnesia ( $\text{MgO}$ ), beryllia ( $\text{BeO}$ ), silicon carbide ( $\text{SiC}$ ), titanium carbide ( $\text{TiC}$ ) and boron nitride ( $\text{BN}$ ). These ceramics consist of crystallites 1 to 50  $\mu\text{m}$  in diameter, produced by hot pressing or sintering of powders. In cryogenic applications  $\text{Al}_2\text{O}_3$  has served as an insulator,  $\text{Al}_2\text{O}_3$  and  $\text{TiC}$  serve as bearing materials, and all of the above could serve as fillers for polymers.

Polycrystals in a glassy matrix include porcelain, pyroceram, and machinable glass ceramic (Corning Code 9658). Porcelain is a common load-bearing electrical insulator at room temperature; to reduce thermal shock, pyroceram may be substituted for cryogenic service.

A promising cryogenic material is the machinable glass ceramic which consists of finely dispersed mica (~50 vol.%) in a boro-alumino-silicate glass matrix [161]. Most ceramics are unmachinable, but this material can be machined to precise tolerances using common metal-working tools. The mica dispersoids cleave easily, aiding chip formation and conferring machinability approaching that of a plain carbon steel. The material is thermal-shock resistant, fully dense, inert, and insulating; it has a thermal expansion comparable to metals.

Cermets are ceramic-metal composites where the metal serves as the matrix or filler. Sintered titanium or tungsten carbides bound with cobalt or nickel are used as bearing materials for cryogenic axial mechanical pump seals to take advantage of their high hardness and wear resistance.

#### c. Mechanical and Physical Properties

The mechanical behavior of ceramics at room temperature is reviewed by Wachtman [162], but cryogenic data are available for few materials, and ceramics are ignored in most cryogenic handbooks. Alumina is representative of the glass-free ceramics. Crystal size, purity, and density affect the material properties. The tensile strengths for

commercial grades of alumina range from 155 to 310 MPa at 295 K. The strength increase at 77 K for 96% grade alumina amounts to about 35% [163]. The fracture toughness of alumina is low (4.3 to 5.2 MPa·m<sup>1/2</sup>) at all temperatures between 300 and 77 K [164]. The elastic and shear moduli are significantly higher ( $E = 398$  GPa at 295 K) compared with steel, and all elastic properties are regular functions of temperature [165].

High thermal conductivity is an outstanding advantage of some glass-free ceramics. Beryllia (BeO) has the highest thermal conductivity of any oxide ceramic, with 300 K values for dense polycrystals approaching 310 W/(m·K) or 75% of the value for copper. For this reason, BeO is added to thermal contact greases for cryogenic applications. Other thermally conductive ceramics are SiC, BN, and AlN, which have 300 K thermal conductivity values of 250, 300, and 660 W/(m·K), respectively. Temperature reduction to 77 K has only a slight effect on the thermal conductivity [166].

Selected cryogenic data for cermets are shown in Table 7.2 [167].

#### d. Design

Ceramics are preferably used in compression. Tensile strength and toughness is low in all ceramics. Scatter in properties is high, and polyaxial stress conditions further reduce tensile strengths [168]. If tensile stresses are unavoidable, all sources of stress concentration must be minimized or eliminated.

For optimum mechanical properties, fully dense, fine grained, high quality ceramics should be chosen and carefully handled. Flaws are often introduced inadvertently owing to abrasion or thermal shock caused by rapid cooling. These flaws should be removed by polishing, and the finest possible surface finish is desirable.

For most ceramics, it is assumed that the flexure strength is twice the tensile strength. In critical cases, any assumptions should be verified. Proof testing is recommended to overcome uncertainties. The most sophisticated design approaches combine statistical analysis with proof testing [169, 170].

### 7.4. Polymers

Polymers are chain-like molecules in amorphous or semicrystalline arrangement, having strong covalent intramolecular bonds and weak intermolecular bonds. Numerous structures are possible, and the great diversity of commercial products now available defies any rigorous classification. The conventional classification distinguishes between thermoplastics and thermosets. Thermoplastics have huge chain structures with little or no crosslinking; they soften and can be molded or shaped repeatedly if heated. In contrast, thermosets are strongly cross-linked, and they cannot be reshaped after the initial curing. Beyond this, material designations are confusing. Thousands of structural variations may be categorized by a single generic name, and multiple tradenames exist for similar products. A useful aid is the list of trade designations compiled by Titus [171].

#### 1. General Information

Polymers offer moderate cost, moldability, machinability, low friction, desirable insulating properties, and, in some cases, transparency. Unfortunately, bulk applications at 77 K are severely limited owing to large thermal contraction, low ductility, and poor thermal shock resistance. This leaves miscellaneous uses that are discussed separately in various sections of this report: adhesives (10.1), sealants (10.2), gaskets (11.3.1, 11.3.2), coatings (12.3.6), windows (7.2.2), potting compounds (10.3), fibers (8.3.), films (8.2.2.) and foams (8.1.). The cryogenic properties and uses of polymers are comprehensively reviewed by Landrock [172]. Additional design data can be obtained from NBS Monograph 132 [17].

One parameter limiting cryogenic utility is the glass transition temperature,  $T_g$ , defined as the switch-over point between rubbery and glassy behavior. All polymers have a  $T_g$  value higher than 77 K. Consequently there are no cryogenic elastomers, and the 77 K

Table 7.2. Some properties of cermets.

Material	Flexural strength GPa		Charpy impact energy J		Mean thermal expansion coefficient $\times 10^{-6}/K$	
	@295 K	@77 K	@295 K	@77 K	77 to 295 K	295 to 400 K
WC/Co	2.05	2.16	1.35	1.35	1.87	2.52
WC/Ni	2.32	2.52	----	----	1.82	2.52
TiC/Ni, Mo	1.78	1.79	0.34	0.27	2.08	3.70

Note: 1 GPa = 0.145 ksi.  
 1 J = 0.737 ft·lb.  
 1 K = 1.8°F

ductility of polymers is low. Upper service temperature limits are also encountered, owing to thermal softening or decomposition. Elastomers and adhesives are two polymer classes that lose strength rapidly above 340 K. Upper service temperature limits for polymers are listed by Wood [173].

Nitrogen at low temperatures is deleterious to polymers. PTFE and five other thermoplastics have exhibited crazing (porosity and fibrillation) and/or reduced fracture stresses when tensile tested in cold liquid or gaseous nitrogen, as compared to vacuum or helium environments [174-176]. Deleterious effects on mylar are observed in gaseous nitrogen between 50 and 160 K, where the fracture stresses are reduced by up to 25%, compared to vacuum [176].

b. Specific Materials

For most cryogenic applications fluorocarbons are the recommended thermoplastics and epoxies are the recommended thermosets.

Fluorocarbons. The most widely used fluorocarbons for cryogenic service are the Teflons--polytetrafluoroethylene (PTFE) and fluorinated ethylene propylene (FEP). These materials are recommended for gaskets and seals (10.0), insulation (13.5.1), antifriction devices (12.3), and protective coatings.

Both PTFE and FEP are chemically inert and thermally stable between 77 and 367 K. PTFE has a lower friction coefficient and is harder and more wear resistant. FEP offers processing advantages. The most serious disadvantage of these fluorocarbons is their tendency to cold flow under static loading, particularly at higher temperatures. If this is a problem, stronger fluorocarbons may be substituted, such as polychlorotrifluoroethylene (PCTFE).

Epoxy resins. Resins are thermosetting polymer systems containing highly reactive three-membered rings of oxygen and carbon which form cross-links when mixed with curing agents. The most common epoxy resin is diglycidal ether of bisphenol A, for which there exist hundreds of curing agents, flexibilizers, modifiers, and fillers.

Despite numerous variations all epoxies are classified as three types - rigid at 295 K, semiflexible at 295 K, and flexible at 295 K. Rigid types are strong at 367 K, but brittle at 77 K [177]. Highly flexible types resist embrittlement at 77 K, but soften excessively at 367 K. Therefore semiflexible types may be best for general use in NTF applications in the range 77 to 367 K. The selection of epoxy adhesives (10.1), sealants (10.2), potting compounds (10.3), and composite matrix materials (9.2) are discussed in the sections noted.

c. Mechanical and Physical Properties

Cryogenic temperatures produce large property changes in polymers. General trends for a temperature reduction from 300 to 77 K are [172]:

tensile, compressive, flexural strength	}	increase
fatigue resistance		
Young's modulus		
creep resistance		
thermal contraction		
electrical resistance		
adhesive strength	}	variable
thermal conductivity		
ductility	}	decrease
toughness		
impact resistance		
thermal expansion coefficient		
specific heat		



The tensile properties of bulk thermoplastics are compared in Table 7.3 [178-179]. Ultimate tensile strengths range from 8.5 to 149 MPa (1.3 to 22 ksi) for the polymers shown. Elongation at break is 7.5% for PTFE at 77 K, and lower for other polymers. Young's moduli at 300 K for 67 polymers range from 26 to 38 MPa, and, despite a large stiffness increase at 77 K, polymers remain relatively low stiffness materials. Thermal contractions for polymers are large (Fig. 3.10). The thermal conductivities of amorphous polymers show a steady decrease at low temperatures, with values of 0.16 to 0.22 W/(m·K) at 77 K. Semicrystalline polymers show a mild decrease or a mild increase at low temperatures with values between 0.1 and 1 W/(m·K) at all temperatures between 350 and 77 K [180].

### 7.5. Filled Polymers

Powders or chopped fibers may be added to polymers prior to molding or casting to achieve improved mechanical and physical property combinations. The designer can purchase filled polymers or fabricate his own. Filled polymers, like fiber-reinforced laminates, are composite materials. But laminates achieve superior strengths. The primary goal of a filled polymer is usually increased thermal conductivity or decreased thermal contraction, with perhaps a moderate improvement of strength. Thus, the thermal conductivity of epoxy can be increased by a factor of 10 by adding powdered aluminum, or total contraction between 295 and 77 K can be reduced from 1.25 to 0.4% by the adding 75% glass filler. Fillers for specific purposes are listed in Table 7.4.

Cryogenic uses for filled polymers include antifriction components, ball-bearing separators, spacers for superconducting magnet windings, and reinforced gaskets and valve seats. Guides to predicting engineering properties based on filler composition are provided by several authors [181-187].



Table 7.3. Tensile properties of bulk thermoplastics.

Material	Temp. K	Glass transition, $T_g$ , K	Ultimate strength, MPa	Elong., %	Youngs modulus, MPa
Polymethylmethacrylate PMMA	300 77	377	76 122	9.4 4.0	1274 3138
Polystyrene PS	300 77	373	30.1 41.2	2.6 2.3	1274 1863
Polyvinylchloride PVC	300 77	355	58 149	139 6.1	980 2350
Polypropylene PP	300 77	255	33 89	853 4.7	686 2450
Polytetrafluoroethylene PTFE	300 77	223	18.5 37	530 7.5	402 1370
Polyethylene PE	300 77	164	8.5 108	487 4.3	598 2450

Note: 1MPa = 0.145 ksi

Table 7.4. Epoxy fillers for polymers.

Purpose	Filler
Reduced contraction	glass, metals, ceramics,
Improved impact	chopped glass, asbestos, aluminum, fluorocarbon fibers
Abrasion resistance	glass, alumina, iron, sand, silicon carbide
Improved thermal conductivity	aluminum, copper
Reduced thermal conductivity	glass
Improved electrical conductivity	aluminum, copper, silver
Machinability	aluminum, copper
Extenders	clay, silicates, metal salts
Reinforcement	asbestos, glass, wire, fibers
Lubricity	graphite or $\text{MoS}_2$

## 8. FOAMS, FILMS, AND FIBERS

This chapter discusses films, fibers, and foams that offer unusual properties owing to unique aspects of material form, size, or structure.

### 8.1. Foams

Foams have a porous, cellular structure that makes them useful thermal insulators by virtue of their lower thermal conductivity and lower density compared with solid materials. Polymer and glass foams are available. Polymer foams are easily cut, and they conform readily to container shapes. Glass foams are stronger and more rigid.

The average thermal conductivities of six unevacuated foams between 300 and 77 K range from 0.033 to 0.055 W/(m·K) [188]. Evacuated foams offer still lower thermal conductivities. The thermal contractions of polymer foams between 300 and 77 K are typically 0.8 to 1.8 %. Tensile strengths between 77 and 350 K may be as high as 0.35 MPa [172]. Polyurethane foam retains useful strengths at these temperatures. Sparks [189] provides an extensive review of the available information on polyurethane and polystyrene foams.

### 8.2. Films

Films are used for electrical insulation, linings, and vapor barriers, often in combination with foams. Desirable films combine high strength and strain capability with flexibility and insulating properties. High strength enables a reduction in film thickness with resultant increased flexibility.

Commercial films having good electrical insulating properties and acceptable for cryogenic service are listed in Table 8.1. Polyethylene terephthalate (PET) is used for cryogenic diaphragms, expulsion bladders, and tank liners. Aluminized PET serves as a thermal radiation shield for insulation systems. At 77 K PET retains 5% elongation at break, an elastic modulus of 100 GPa, and a tensile strength of 303 MPa. Van de Voorde [178] indicates that polyimide (PI) film is even stronger than PET, but both retain useful mechanical properties between 77 and 367 K [179, 190].

Javorsky and Popelis [191] present thermal contraction data which are useful to match films and foams with various substrates. Additional data are presented by Landrock [172] and Jelinek and Muller [192]. For extremely low thermal contraction, glass cloth/polymer film laminates are recommended.

### 8.3. Fibers

Owing to small size and relative freedom from manufacturing defects, fibers are effective reinforcements for composite laminates and filled polymers. Since fibers contain fewer flaws compared to materials in bulk form, fibers approach theoretical strengths more closely than do bulk materials. Size effects also confer higher strain capability for fibers. For example, bulk glass fails at 0.1% tensile strain, whereas some glass fibers are capable of 5% strain.

In the order of value as cryogenic materials, the fibers may be ranked as follows:

- 1) glass fibers,
- 2) advanced fibers (graphite, boron, kevlar),
- 3) polymers,
- 4) metals, and
- 5) ceramics

For structural composite laminates requiring strength or stiffness, glass and advanced (high modulus) fibers are of greatest technological importance. Glass and advanced fibers are poor conductors of heat, and they exhibit low thermal contractions, which improve the performance of composites using epoxy matrices.

Table 8.1. Tensile properties of polymer films.

Material	Temperature	Tensile strength MPa	Elongation %	Young's modulus GPa
Polyimide	298	178	44	5.00
PI	77	318	16	6.27
Polycarbonate	298	63	112	
PC	77	132	4.5	
Polyamide	298	81	21	
PA	77	142	3.5	
Polyethyleneterephthalate	298	162	88	4.41
PET	77	255	9.5	8.53
Polyvinylchloride	298	22	156	1.08
PVC	77	93	3.6	2.11
Polyethylene	298	24	525	
PE	77	103	4.3	
Polytetrafluoroethylene	298	20	480	
PTFE	77	42	6.5	

Note: 1 MPa = 0.145 ksi

In most cases, fibers are manufactured and used as coated strands (bundles of fibers). The coatings prevent surface damage and enhance compatibility with specific matrix materials. Costs are generally high for quality fibers, and prices vary with weave, yarn, and finish types. The production, availability, price, and future trends of advanced fibers are discussed in detail by Kaiser [193]. The following discussion is taken from Kasen [194-200].

Glass fibers Glass is the most widely used cryogenic fiber. Advantages are high tensile strengths up to 3.7 GPa, low cost, availability, low thermal and electrical conductivity, and low thermal contraction. The most serious disadvantage is low elastic modulus (73 to 86 GPa). The most common glass finish is silane A, which is compatible with polyester, epoxy, and phenolic matrices.

The recommended glass fiber types are E-glass and S-glass (section 7.2.). Electrical insulating grade E glass is a general purpose fiber costing about \$1/kg for roving (bundles of twisted strands). Cameron [145] reports the breaking strengths of E glass fibers to be 3.44 GPa at 295 K and 5.65 GPa at 77 K. Specialty grade S-glass is a higher strength fiber, about 30% stronger than E glass. Two alternatives, S-901 and S-2 glass fibers have similar mechanical properties, but S-2 glass is cheaper, costing twice as much as E glass.

Graphite fibers Graphite fibers rank a distant second to glass in total cryogenic usage. Graphite fibers are electrically conductive, extremely stiff, and brittle. Costs are moderate to high. There are three graphite fiber types:

Type I - lowest strength, highest modulus, highest cost

Type II - intermediate strength, modulus, and cost

Type III - highest strength, lowest modulus, lowest cost

For Type III fiber, strengths of about 2.6 GPa are available with a modulus of 225 GPa. For Type I fiber, moduli of about 520 GPa are available at strengths of 1.9 GPa. Thus, the highest graphite fiber strengths are comparable to E glass, but the highest modulus is 7 to 8 times greater than E or S glass.

Continuous or chopped graphite filaments, fiber flock, and woven cloths are available, mostly produced from the pyrolytic degradation of polymers. Typically, fibers of about 8  $\mu\text{m}$  are bundled in strands of 1000 to 160,000. Costs range from tens to hundreds of dollars per kg. One manufacturer sells low priced pitch-base fibers, but the mechanical properties are inferior.

Kevlar 49 The aramid polymer fiber Kevlar 49 may be considered as a replacement for glass if a 45% higher fiber modulus and 42% lower fiber density is needed. Of all fibers, Kevlar 49 offers the highest possible strength-to-density ratio. It offers a high negative thermal contraction coefficient. However, Kevlar 49 is restricted in use to epoxy resin matrices, and compressive properties are poor. Prices range from nearly equivalent to S glass to seven times higher.

Boron Boron fibers may be considered if both high strength and high stiffness are required. Boron is as strong as S-glass, but five times stiffer. The elastic modulus at 295 K is 410 GPa, twice the value of steel. Boron also offers extremely high compressive strength. Unfortunately, owing to a low bend radius, Boron is unsatisfactory for complex shapes. Another deterrent is high cost (hundreds of dollars per kg).

Polymers Polypropylene, polybenzimidazole, nylon, and other synthetic fibers were studied to identify relatively strong cryogenic fibers having minimal thermal conductivity [172]. PET fibers can be recommended as useful insulation for electrical conductors since the strands are flexible enough to bend around small radii. Flexible polymer fibers have also been used as thermal insulation for cryogenic pipes and transfer lines, and nylon finds applications in expanding, inflatable bladders.

Metals Metals are the least important reinforcements for composites because metal strengths and moduli are low, while densities and thermal conductivities are higher compared to glass and the advanced fibers discussed above. Nevertheless, metal "fibers" for molded



plastic reinforcement may be formed by cutting fine alloy wires. Stainless steel wires in diameters as low as 19  $\mu\text{m}$  are readily available.

Ceramics Alumina, silicon carbide, and boron nitride fibers are in developmental stages. Availability is limited and costs are high. The main advantages of ceramic fibers are their insulating properties at very high temperatures. There are no cryogenic applications.

## 9. STRUCTURAL COMPOSITE LAMINATES

Composite laminates are epoxy or metal matrix materials reinforced with continuous uniaxial fibers or woven fiber clothes, with fiber volume fractions of about 50 to 70%. These laminates are capable of high strength-to-weight, strength-to-thermal conductivity, and stiffness-to-thermal conductivity ratios which cannot be matched by structural alloys. But the use of composites at cryogenic temperatures is hampered by a lack of service experience, standards, and design data for specific composites. Material costs and property variability are higher, while design procedures and joints are more complicated with composites than with structural alloys.

Most laminates are linear-elastic when loaded to failure, with tensile strains ranging from very low in transverse fiber orientations to as high as 5% for some glass reinforced laminates in the longitudinal fiber orientation. A peculiar failure mode occurring in fatigue is wear-out, which is a process of fiber/matrix debonding associated with stiffness reduction. Design with composites is thus more difficult than with structural alloys, which are far less anisotropic.

There are no established procedures or standards for design with composites at cryogenic temperatures. The initial approach should be based on procedures in the Advanced Composites Design Guide [201] and MIL HDBK 17A [202], with Kasen's reviews [194-200] serving as a guide to cryogenic property trends. Macromechanical theory provides a basis for predicting the mechanical properties of proposed layup configurations. Quality assurance is necessary in critical structural applications to account for material variability, which strongly depends on workmanship during fabrication.

Glass fiber reinforced epoxy is by far the most widely used composite, accounting for perhaps 90% of all current applications. Cryogenic uses include filament wound aerospace pressure vessels, load-bearing thermal standoffs and supports, dewar construction, and electrical/thermal supports for superconducting magnet windings at temperatures as low as 4 K. Glass-epoxy composites are recommended for applications requiring high tensile strength and toughness, if low stiffness can be tolerated, and if sufficient benefit accrues in performance and weight considerations compared with alloys. Advanced fibers are possible substitutes for glass fibers in epoxy laminates for special purposes, as noted in the text. For discussion, structural composites are classified below as industrial (high pressure) polymer laminates, low pressure polymer laminates, metal-matrix laminates, and selectively reinforced structures.

### 9.1. Industrial Polymer Laminates

Examples of industrial laminates acceptable for cryogenic service are listed in Table 9.1. Industrial laminates are commercially fabricated at curing pressures up to about 8 MPa. They are relatively inexpensive and available in bulk form as plate, sheet, or bar. These materials are recommended for non-critical electrical or thermal insulating purposes at moderate stress levels. Components can be readily machined from bulk stock. As indicated in Tables 9.2 to 9.3, and Fig. 9.1, there is generally a modest improvement in thermal and mechanical properties at cryogenic temperatures, but the existing specifications cover only room temperature properties. Therefore most industrial laminates are not recommended for cryogenic structural applications subject to high stress, unless quality control tests are performed. Cryogenic grades of G-10 and G-11, called G-10CR and G-11CR, have been developed for more critical cryogenic applications, and some data for these newly developed grades are shown in Table 9.2 and 9.3.

### 9.2. Low Pressure Polymer Laminates

Low pressure laminates are fabricated at low curing pressures (from zero to 2.8 MPa). With these laminates, designers can custom fabricate structures in situ, exercising options in fiber and resin selection so as to tailor properties to specific applications. For example, honeycomb structures or composite overlays can be built up using preimpregnated tape or filament winding as described by MIL HDBK 17A [202], and Advanced Composites Design Guide [201].

Table 9.1. Industrial laminates of primary cryogenic interest.

Commercial standard	MIL-P and LP designation	Description
NEMA/ASTM LE	MIL-P-15035, Type FBE	Cellulose fabric, fine weave phenolic resin
NEMA/ASTM C	MIL-P-15035, Type FBM	Cellulose fabric, medium weave, phenolic resin
NEMA/ASTM G-5	--	Glass cloth, melamine resin
NEMA/ASTM G-10	MIL-P-18177, Type GEE	Glass cloth, epoxy resin
NEMA/ASTM G-11	MIL-P-18177, Type GEB	Glass cloth, high-temperature epoxy resin
EH57, EH57	LP 5094	Beechwood veneer, phenolic impregnated

Table 9.2. Mechanical properties of G-10CR and G-11CR industrial laminates [200].

Temperature (K)	Young's modulus, E, (Tensile-W), GPa	Poisson's Ratio	Tensile strength $\sigma^{TU}(W)$ MPa	Compressive $\sigma^{CU}(W)$ strength MPa	Compressive $\sigma^{CU}(N)$ strength MPa	Shear strength $\sigma^I$ (short beam-W) MPa	Shear strength $\sigma^I$ (Gillotine-W) MPa	Strain to failure $\epsilon^{+U}$ %
G-10CR								
295	28.03	0.150	415	375	420	60	42.3	1.75
76	33.73	0.190	825	833	693	131	61.3	3.44
H-11CR								
295	31.99	0.157	469	396	461	72	40.6	1.82
76	37.30	0.223	827	803	799	120	56.5	3.21

Note: 1 GPa = 0.145 msi  
1 MPa = 0.145 ksi  
W = warp direction  
N = normal direction

Table 9.3. Thermal properties of G-10 CR and G-11 CR industrial laminates.

Material/direction		Thermal contraction ( $L_{293}-L_{75}$ )/ $L_{293}$ , %	Thermal conductivity W/(m·K) 295 K      77 K	
G-10CR	Warp	0.235	0.860	0.380
	Fill	0.269	-	-
	Normal	0.730	0.600	0.275
G-11CR	Warp	0.219	0.730	0.315
	Fill	0.243	-	-
	Normal	0.620	0.620	0.275

Note: 1 W/(m·K) = 0.578 BTU/(ft·hr °F)



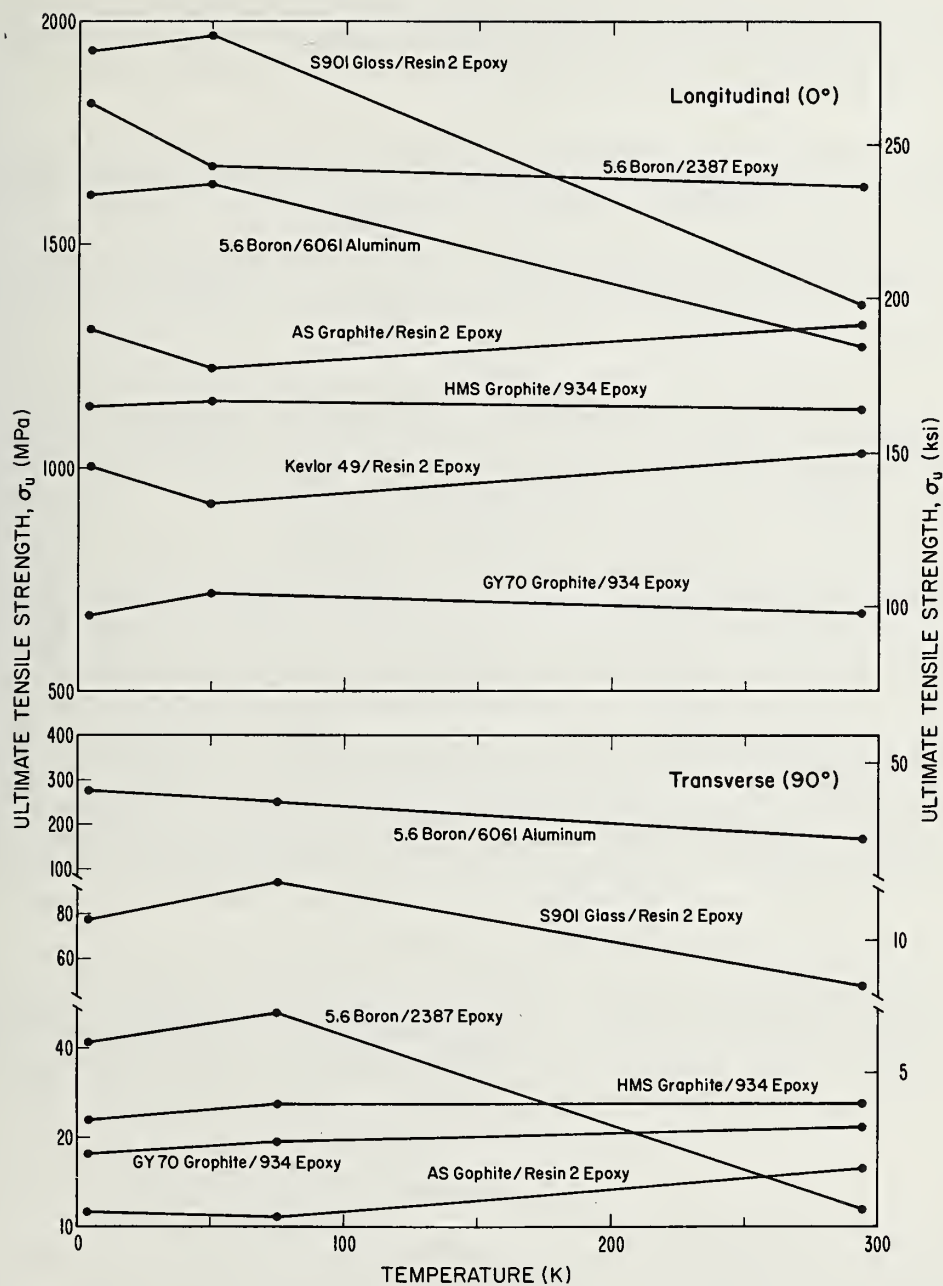


Figure 9.1. Cryogenic effects on the ultimate strength of composites.

Glass fibers are used for high strength, low modulus laminates, whereas graphite or boron fibers are used for high modulus laminates. In either case, epoxies are the recommended matrix materials. The selection of a particular epoxy resin is more important for low modulus laminates than for high modulus laminates since fibers of lower stiffness transfer greater strain to the matrix.

Epoxies suitable for cryogenic applications are discussed by Kasen [199]. For NTF applications at 367 K, highly flexibilized resins such as NASA Resin 2 are unacceptable owing to loss of strength above room temperature. Rigid or semirigid epoxies are acceptable but no single formulation can be recommended as clearly superior. Kasen therefore recognizes two approaches to matrix selection:

- 1) Select a resin system that has been used previously with composites, and for which cryogenic data already exist in the literature. (This method may be fruitless since many tested epoxies are no longer readily available).
- 2) Base initial selection on cost and availability, then fabricate laminates and perform screening tests at the temperatures of interest. Screening tests are recommended in any case, since performance is dependent on fabrication variables, as well as on the choice of materials and layouts.

Based on the materials selected, the laminate properties can be predicted based on the key properties and using macromechanical theory. Representative data for uniaxially reinforced composite laminates are shown in Fig. 9.1. For a description of temperature effects on tensile, compressive, flexural, shear, fatigue, impact and thermal properties, the reader is directed to Kasen [194-200] and Hartwig [203].

### 9.3. Metal Matrix Laminates

Metal matrix composite laminates are in developmental stages. Boron-reinforced 6061 aluminum is the only commercially available metal matrix composite and the only one for which cryogenic data exist. Potential advantages for this material are higher strength-to-density and modulus-to-density ratios compared with structural alloys, such as AISI 304. Compared with boron-epoxy low pressure laminates, boron-aluminum offers no mechanical property advantages in the fiber direction, although tests in the transverse direction show sevenfold superiority in strength and fourfold superiority in modulus. Unfortunately, the fabrication process for boron-aluminum introduces residual stresses that cause nonlinear stress-strain behavior. As a result, the stress-strain behavior of crossply boron-aluminum composites is less predictable compared with boron-epoxy composites. Therefore boron-aluminum is currently used only for uniaxially loaded structures; NTF applications are not anticipated.

### 9.4. Selective Reinforcement

Advanced composites can be bonded to metal components to selectively stiffen or strengthen primary structures [201]. This technique might be applicable in the design of stings or other NTF model supports. For example, graphite or boron-epoxy overlays or cores in stainless steel shafts should result in a stronger, stiffer shaft while maintaining the simplicity of metal shaft connections [194].

This chapter is subdivided into sections on adhesives, sealants, potting compounds, and contact agents, but many of the materials discussed could serve multiple functions.

### 10.1. Adhesives

An adhesive is any substance capable of bonding two materials together. Silicones, varnishes, cements, and other materials are acceptable for light bonding applications, but this section is concerned with structural adhesives where high strength is the primary criterion. A structural adhesive is defined as having a shear strength in excess of 6.9 MPa.

Cryogenic adhesives were originally developed for aerospace applications between 20 and 800 K, for bonding metals that could not be welded or riveted, for bonding insulation or wires, for fabricating honeycomb structures, or to prevent galvanic corrosion between contacting dissimilar metals. Many structural adhesives are now available that serve as well at 77 K as at room temperature, but no single formulation serves every purpose. Although polytetrafluoroethylene is well known for its antisticking property, Eppinger and Love [204] created polytetrafluoroethylene-to-steel bonds as strong as 55 MPa using a nitrile-phenolic adhesive. Thus, it appears that any solid can be adhesively bonded, using the appropriate technique.

Epoxies, modified epoxies, and thermosetting polyurethanes are generally recommended for bonding metals and nonmetals at temperatures in the range 77 to 367 K. Many products are available as solvent based compounds, the solvent being removed by curing at room or elevated temperatures. Epoxies offer the greatest versatility in bonding many adherends, with or without fillers or reinforcements. Epoxy-nylons are stronger and tougher than unmodified epoxies, but more susceptible to moisture-induced degradation, and more sensitive to bond-line thickness. Polyurethanes are stronger in shear, but more difficult to process than epoxies. In any case, the choice of adhesive is influenced by the joint geometry and type of loading; selection should be based on comparison of lap-shear, short-beam-shear, butt-tensile, or peel-test data, according to the type of loading anticipated in service [205].

Regardless of the type of adhesive selected, there are proper techniques for optimizing performance. Adhesion is promoted by cleaning and roughening the adherend surfaces, and the use of primers and couplers may be beneficial. Thin layers of adhesive are generally more resistant to thermal cracking than are thick layers. Peeling resistance is higher if the adhesive surrounds the substrate completely as compared with a local application. Peeling resistance is also higher if the adhesive is constrained in a narrow gap between two components as opposed to being applied open-faced.

To achieve optimum performance with epoxies, the stress concentrations arising from differences in elastic moduli and thermal contraction of the adhesives and adherends can be reduced using fillers or cloth reinforcement [206]. Van de Voorde [178], Hamilton, Greene, and Davidson [207] cite the following examples of commercial filled adhesives with thermal contraction adjusted for specific metals:

- 1) Stycast 2850 FT-X3 matches OFHC copper
- 2) Stycast 2850 GT matches yellow brass
- 3) Stycast 2850 FT matches aluminum
- 4) Stycast 2850 FT + quartz matches AISI 304

More detailed information can be obtained from the literature [203-223]. Commercial epoxy adhesives are discussed by Hartwig [203] and by Mackowski, West, Samsel, Holdeman, and Peters [208]. Hillig, Legrand, Kelran, de Tores, and Coblentz [209] identify the better adhesives for specific adherends and temperature ranges of interest. Froelich and Fitzpatrick [211] evaluate epoxy, varnish, and glass cloth supported adhesives at 77 K, exploring the influence of adherend type and surface preparation. Williamson and Olien [212] provide 130 abstracts from research articles on adhesives and sealants. There is also a welter of manufacturer's product bulletins to consider. The recommended approach is to consult this literature as a guide to the proper adhesive type for a particular application, and then purchase a number of commercial products which can be screened experimentally.



## 10.2. Sealants

Sealants are used in model building to fill holes, joints, and surface imperfections. In cryogenic applications, sealants are also used to waterproof or exclude dirt. Adhesives, solders, platings, and putty-like materials for caulking and fairing are available for cryogenic service, but published data and experience in this area is lacking.

Epoxyes. Araldite XD580 is an example of a putty-like resin which can be finger-formed before it sets up. This resin has a thermal expansion coefficient near that of copper, and it presents no cracking problems when used in magnet coils at temperatures as low as 4 K.

Silicones. The use of RTV silicones as cryogenic sealants is discussed by Screihans and Robinson [213]. Silicones lose their elastomeric properties at temperatures below 200 K, but they retain significant strength and superior peel resistance compared with polyurethanes.

Rubbers. Rubber-based sealants are also useful at cryogenic temperatures. One commercial product is pliable after evaporation of the solvent; it can be smeared on to provide temporary seals, and is easily removed with acetone [214]. Sta Set is good for light bonds between "stick-resistant" Teflons and metals, glass, or other plastics [215].

## 10.3 Potting Compounds

Castable potting compounds may be used to embed instrumentation. Sealing wax is used at room temperature since it can be easily melted and removed. For cryogenic service, epoxyes are recommended. As discussed by Hartwig [203], thermal cracking is a problem in rigid fully reacted epoxy resins, owing to lack of cryogenic ductility. Therefore, semi-flexibilized resins may be best for general use at NTF test temperatures. More experience is needed in this area.

## 10.4. Contact Agents

Contact agents are used to improve energy transfer between heater wires, thermocouples, heat sinks, and electrical contacts. Bond strength is not the primary requirement, and strong adhesive properties may actually be undesirable if quick disconnection of fragile elements is necessary.

Any material with wetting properties can be considered for contact applications, but the most practical choices are solders, adhesives, varnishes and greases. Varnishes and greases are discussed below; solders and adhesives were discussed in sections 5.2. and 10.1, respectively.

### 10.4.1. Varnish

GE varnish 7031 is a common room temperature electrical insulating thermal contact varnish and it is often used for cryogenic service. This particular varnish is favored partly by tradition, and there are no published data comparisons with other commercial varnishes.

GE varnish 7031 can be used at all temperatures between 77 and 367 K. Varnish is more strongly adhesive than greases, yet more easily removed than epoxy adhesives. Commercial varnish contains a solvent which, if allowed to air dry, leaves a flexible resin which can be softened and removed by soaking in methyl alcohol. Baking produces a thermoset which is as difficult to remove as epoxy adhesives.

The thermal cracking resistance of varnish at cryogenic temperatures in thin layers compares favorably with adhesives. The thermal conductivity of the varnish is nearly equivalent to Teflon [219]. Peeling resistance is higher than some well-known epoxy adhesives [217] but lap shear strengths are reported to be lower, with representative values of 2.71 MPa at 293 K and 2.04 MPa at 77 K [211].

#### 10.4.2. Grease

Greases are typically used as thermal contact agents or as release agents for gaskets and seals. Greasing a specimen/heat-sink interface may increase the heat flow rate by up to a factor of ten, but the extent of improvement is difficult to predict. Some thermal properties for Apiezon N and T greases are reported at cryogenic temperatures [220]. Due to freezing, some mechanical strength is developed at cryogenic temperatures, but lack of strength at room temperatures limits some applications.



## 11. SEALS

Seals prevent fluid flow between coupled components for the purpose of maintaining pressure differences or simply to separate gases or liquids. Nondemountable seals are created by welding, brazing, soldering, or adhesive bonding. Demountable seals are created by pressing a deformable sealing material against couplings to block the interface.

### 11.1. Nondemountable Seals

Adhesives and alloys for brazing and soldering are discussed in section 5. Thermal expansion or contraction behavior should be considered when sealing by these methods. For example, bismuth alloys are unique for soldered seals because they expand on solidification, and will not shrink away from the seal interfaces [224]. Thermosetting resins can also be used, if thermal cracking can be avoided. To minimize thermal cracking, the expansion coefficients of the resin and coupling materials must be closely matched (see section 10.1.).

### 11.2. Demountable Seals

Demountable seals for cryogenic service are reviewed by Robbins and Ludtke [225] and Daniels [226]. There are two types: static and dynamic. A static seal is one where there is no relative movement between the seal interfaces during operation. Shifting of the interfaces during cool-down or otherwise may break the intimate contact of a static seal and should be avoided. Dynamic seals are most often of the spring type discussed below.

#### 11.2.1. Design Considerations

The most familiar static demountable seals are O-ring or gasket seals. In addition there are temperature-, spring-, or pressure-activated seals, and toggle and boss seals. Cryogenic sealing by any of these methods requires accounting for some factors not considered in room temperature applications:

- 1) Limited reusability of the seal material,
- 2) Failure owing to thermal shock,
- 3) Distortion of machined interfaces, owing to thermal gradients, and
- 4) Seal separation owing to differential thermal contraction.

Thermal contraction, causes difficulties in sealing dissimilar materials. Nevertheless, effective seals between diverse materials, including glass-to-metal, metal-to-plastic, and plastic-to-glass combinations can be obtained using appropriate techniques [227-267].

Numerous seal designs for specific applications are cited in the literature. Since a detailed illustration of all possibilities is not possible here, Daniel's [226] approach to seal design is recommended: (1) determine the performance requirements, (2) screen the existing sealing concepts or develop new ones, and (3) evaluate the alternatives in terms of cost, maintenance, and availability of materials.

#### 11.2.2. Gasket and O-Ring Seals

Gaskets and O-rings must be compressed between relatively heavy flanges using a ring of tightening screws. Effective seals are thereby obtained, but flanges are undesirable for some applications owing to space, weight, and load considerations. Alternatives are discussed in sections 11.2.3. and 11.2.4.

Gaskets are rectangular in cross section and, therefore, stable when positioned on flat flanges. But flat flanges require accurate machining, since scratches or lack of planarity may cause leakage. O-rings are circular in cross section, and they seal over a narrower contact area, requiring less force than gaskets. Grooved or tongue-and-grooved flanges are used to maintain symmetry as the flange is tightened. Often the groove is

made smaller in volume than the O-ring, so the latter will be partially extruded from the groove, improving the seal. Teeth opposite the groove will improve stress concentration so that tightening loads may be reduced. However, metal O-rings or putty-like materials may become embedded and difficult to remove from grooved flanges. A flat aluminum spacer may serve as an alternative to a groove if it is desirable to avoid more expensive machining operations on the flange.

Most seal designs are custom made, requiring experimentation to prove effectiveness. O-ring and flange materials must be compatible since the seal must function as an integrated whole. If dissimilar flange metals are used, corrosion must be avoided by choosing metals closely ranked on the electromotive series, or by using nonmetal O-rings or gaskets. The O-rings and gaskets must not be harder than flange materials, or the flanges will be scratched, causing leaks. If glass is involved, scratches may cause fracture when sealing loads are applied.

Flange surface finish is sometimes critical. Finishes generally range from 8 to 64 microinches, RMS. The flanges should be cleaned with solvents such as acetone or alcohol, and if cold-welding (see section 12.3) is relied upon to aid the seal, assembly should be performed using gloves. Hold-down screws should be tightened gradually and systematically, and thermal shock can be reduced by slow cool-down or filling rates.

Once in operation at low temperature, most static seals function well for extended periods, but some seals fail gradually with temperature cycling. The leading cause of failure is relative motion between the flanges, causing extrusion of the soft sealing materials or coatings.

#### 11.2.3. Temperature-Assisted Seals

Differential thermal contraction provides one basis for effective seals. Consider the design of a nylon collar around a brass component: on cooling, nylon contracts more than brass, and the choking effect creates a seal. A similar effect occurs if aluminum rings or casings encircle steels or other alloys that contract less at a given temperature. If two metals are involved, Teflon or soft metal coatings may be applied to the interface to improve the seal. Stearns [236], Agarwal and Betterton [251] and Vesel [252] describe thermally assisted seals that function at pressures up to 20 MPa.

#### 11.2.4. Spring and Pressure-Assisted Seals

Spring seals are aluminum, stainless steel, or superalloy backbones with soft metal (silver) or plastic (PTFE) coatings. The metal backbone provides sealing forces when compressed, and compensates for dimensional changes during cool-down. The soft coatings improve sealing characteristics. Spring seals are commercially available in a wide variety of geometries such as "K," "C," or "W" cross sections. These seals are simple, readily available, and suitable for dynamic applications. Heavy flanges are not required. Problems include cold flow of soft coatings and accurate load selection.

Pressure-assisted seals are high performance spring seals having an open geometry (for example, C shaped cross section) so that the sealing forces can be increased by pressurization from one side. Seals of this type are used in aerospace engines at temperatures from 20 to 800 K and at pressures up to 435 MPa. Since spring seals are adequate for NTF pressures, the use of pressure-assisted seals is not anticipated.

#### 11.3. Seal Materials

Elastomers, fluorocarbons, hard polymers, and metals are common O-ring, gasket, and coating materials. Soft metals and fluorocarbons are the most common choices for cryogenic service. At room temperature, elastomers, metals, and polymers are all very useful, but there may be some loss of resiliency at elevated temperatures where flow is easier.

### 11.3.1 Elastomers

Elastomers are generally the best materials for O-rings and gaskets at room temperature where they are easily deformed, resilient, and reusable. However, every known elastomer shatters like glass at 77 K. In fact, 200 K is sufficient to embrittle most types [253]. Nevertheless, elastomer O-rings do find some cryogenic applications. They can be used if the seal is formed at room temperature, highly compressed, cooled, and not further stressed at cryogenic temperatures. The seals remain effective at cryogenic temperatures only if the elastomer is highly compressed at room temperature; compression ratios approaching 80% are necessary to build up the restoring forces needed to compensate for the large thermal contraction of elastomers relative to flanges [254-257].

Elastomers that have been evaluated for cryogenic use include molded rubber or RTV silicones, natural rubber, nitrile rubber, neoprene, and others [239, 256, 257]. In one application, an elastomer O-ring provided a simple reusable seal for the lid of an evacuated cryostat [258]. In other applications, elastomer seals can withstand more than an hour of high frequency vibrations at 6.9 MPa and at 20 K, with leak rates less than  $2.5 \times 10^{-3}$  cc, at STP, of helium gas per mm of seal [254].

### 11.3.2. Other Polymers

Polymers work best as flat gaskets cut from sheet. An advantage of polymer gaskets is that they prevent corrosion by avoiding contact between dissimilar metals. However, polymers are harder than elastomers at room temperature. To reduce sealing forces, thin gaskets are used in compression between one carefully machined flat flange and another flange having a semi-circular machined ring. One successful design uses only 7J (5 ft-lb) torque on six 1/4-28 bolts for a 10 mil PET sheet compressed 3 mil using a 2.54 cm diameter ring; leak rates approach those for elastomer O-rings [254].

PTFE (soft, inert, and stick-resistant) is most widely used polymer gasket. Teflon's flow properties are desirable for some applications where harder polymers would simply rupture. On the otherhand, Teflon's cold flow can result in extrusion or compression set, causing leaks. This can be overcome using filled or laminated composite gaskets [225].

### 11.3.3. Metals

Metal O-rings are commonly used at cryogenic temperatures where brittle elastomers would be inadequate. Metals can be directly substituted for elastomers in most seal designs, but resiliency and reusability are sacrificed.

Metal O-rings can be purchased or fabricated. Material selection should be based on mechanical properties, cost, availability, corrosion resistance, and thermal properties. Any soft, ductile, fcc metal or alloy might be considered. Some specific applications are aluminum [259], copper [245], gold [260], indium [261-265], solder alloys [244], and stainless steel [241].

Hardness is one of the primary factors influencing material selection. Soft alloys seal with less force. For this reason harder alloy O-rings such as 304 stainless steel are frequently plated or coated with soft metals (gold, silver, indium) or Teflon. Noble metal platings also provide corrosion protection. All of the metals noted above can be used at cryogenic temperatures down to 77 K, but an upper temperature limit may be imposed by the lower melting points of some metals.

The most versatile cryogenic sealing metal is indium, which is well suited for cryogenic gasket, O-ring, or coating applications. Indium is extremely soft (it doesn't scratch glass), readily available, and inexpensive compared to other corrosion resistant metals. Indium O-rings are easily formed from wire, gaskets are easily cut from sheet, and coatings are easily plated, soldered, or foil wrapped [265-266]. The strong wetting and cold-welding tendencies are useful in applications relying on adhesion to improve the seal [264]. But in some cases, cold welding only makes demounting more difficult and



prevents reuse. Then, greasing the O-ring will facilitate removal [263]. Since indium is soft, flange surface finishes are less critical. A 32 flange finish is generally acceptable [265].

Once tightened, indium O-ring seals can be cycled hundreds of times between 295 and 4 K [264]. The seal holds if there is no extrusion of indium. If extrusion does occur, it may be due to the differential thermal contraction and expansion of dissimilar flange materials [266]. In such a case, intermittent tightening will maintain the sealing force, but ultimately a seal failure is expected [261].

## 12. FRICTION, WEAR, AND LUBRICATION

Friction is the resistance to movement of sliding surfaces. Wear is the resultant surface degradation and material loss that occurs owing to mechanical interaction. Although friction and wear are closely associated, there is no direct relationship. The conditions minimizing friction for a given sliding couple do not necessarily minimize wear.

Wear is in fact poorly understood. Few "laws" have been formulated, and knowledge rests largely on empirical observations. Usually, wear decreases with decreasing temperature or with increasing surface smoothness and hardness. However, the wear rates of materials show much greater variability than their friction coefficients, and there are no measurement standards.

Friction is treated by Bowden and Tabor [268, 269]; Bisson [270] reviews cryogenic behavior. The actual contact area between two sliding bodies is much smaller than the apparent surface areas. All surfaces are rough on the microscale, so true contact occurs only at the meeting of asperities (high points). Friction results from: (1) the shearing of junctions formed by contacting asperities, and (2) the deformation or "ploughing" of the softer material by the harder. These processes evolve heat, particularly at high pressures or high sliding velocities.

Although friction is sometimes desirable, more often it is a problem that must be overcome by force and/or lubrication. Lubrication consists of establishing an interface of low shear strength between the moving parts. In addition to reducing frictional drag, lubricants may also serve to carry away heat, exclude dirt, or prevent corrosion. In thick-film fluid lubrication there is no asperity contact and wear is minimized. At cryogenic temperatures, however, we are usually concerned with boundary lubrication, where the asperities are in contact but the junctions are surrounded by a solid lubricant.

### 12.1. Design Considerations

The lubrication of shafts, cams, gears, rollers, and electrical contacts in the inert cryogenic gases may be more difficult than in conventional air environments, since:

- 1) Austenitic stainless steels, generally favored for cryogenic cryostructural applications, have poor friction and wear properties.
- 2) Metallic cold welding is encouraged in environments that exclude oxygen,
- 3) Practical cryogenic liquid lubricants do not exist, and
- 4) Graphite fails to lubricate properly in certain inert environments.

The selection of a method to reduce friction should be based on the performance requirements of the specific application. A large number of variables affect friction and wear results, among them: sliding velocity, load, chemistry of the environment, type of motion (continuous or intermittent, sliding, rolling, etc.), part geometry, material composition, hardness, finish, and cleanliness. Since reported friction data usually pertain to very specific conditions, preliminary testing may be necessary to verify a choice of materials if friction problems are critical.

Particular consideration must be given to the environment, which often has greater influence on lubricity than temperature has. Friction and wear studies are typically conducted by sliding a loaded rider against a rotating disc. To date, most cryogenic tests have been conducted in vacuum, gaseous nitrogen, liquid nitrogen, or liquid hydrogen environments. Vacuum, gaseous nitrogen, and liquid nitrogen environments are generally considered inert, but the liquid provides a superior cooling medium, the effects of which may be difficult to predict.

### 12.2. The Necessity of Dry Lubrication

Oils and greases are the most common lubricants at 295 K. But oils and greases freeze at temperatures between 220 and 250 K, becoming unusable, glass-like solids. Cryogens, such as liquid nitrogen, also fail to lubricate effectively because their viscosities are orders of magnitude too low. Apparently, the fluorinated polyesters are the



only liquids that do have lubricating properties at cryogenic temperatures [271], but their use requires cumbersome ancilliary cooling and containment systems. Thus, liquid lubrication at cryogenic temperatures is generally dismissed as impractical. Dry lubrication and design using antifriction materials are the usual alternatives.

### 12.3. Friction Coefficients

Figure 12.1 compares the self-friction coefficients of selected materials tested in inert environments. The friction coefficients of metals are relatively high compared with non-metals. The friction coefficients for metals and alloys at 295 K typically range between 0.4 and 1.0. The high friction is also associated with poor wear resistance. Both result from cold-welding.

Cold-welding (or galling) denotes the strong adhesion that occurs when unfilmed metals come into contact. Normally, metals form surface oxide films that prevent cold-welding. These protective films deteriorate by abrasion during sliding, and they cannot reform in environments where oxygen is excluded. Under these conditions, cold-welding is encouraged, possibly leading to the seizure of moving parts. Thus, metallic friction is greater in vacuum than in air and greater in liquid nitrogen than in liquid oxygen. For similar reasons, metallic friction in the gaseous nitrogen NTF environment should be more severe than in conventional wind tunnel environments using air.

#### 12.3.1. Unalloyed Metals

The friction coefficients for 18 metals in inert environments between 4.2 and 600 K are reported by Simon, McMahon, and Bowden [272], Burton, Russell, and Ku [273], and Bowden and Childs [274]. Our knowledge of metallic friction at cryogenic temperatures is based primarily on the findings of these three studies. There is general agreement that metallic friction in inert environments is weakly temperature dependent. The kinetic friction coefficients are usually about 10 to 16% lower than the static coefficients. Ductile metals usually exhibit high coefficients, with values possibly approaching 5 or 10, whereas the coefficients of brittle metals are significantly lower and seldom exceed 1.0. Dissimilar metal couples generally produce lower friction than similar metal couples, and hard riders sliding on soft discs usually give higher friction than soft riders sliding on hard discs.

#### 12.3.2. Alloys

Friction and wear data for structural alloys at cryogenic temperatures are rare. Apparently, steels are the only structural alloys that have been investigated. Simon et al. [272] report friction coefficients for a 0.16% carbon steel and an austenitic stainless steel; Wisander, Maley, and Johnson [275] report friction and wear data for AISI 304 and 440-C stainless steels and SAE 52100 bearing steel at 77 K. All of these steels exhibited high friction coefficients (nearly 0.4 or greater) at 77 K, and the three steels that were wear tested exhibited cold welding and tearing at 77 K.

Since steels are preeminent structural materials, a convenient means of reducing friction would be welcomed. Accordingly, attempts have been made to reduce metallic friction by means of surface diffusion coatings and plating operations that change surface properties.

Diffusion Coatings Simon et al. [272] showed that case-hardening lowers the friction coefficient of a 0.16% carbon steel from 1.3 to 0.6 at 77 K. Hoping for a similar result, Wisander and Johnson [276] applied commercial nitriding, chromizing, and sulfurizing treatments to AISI 304 stainless steel. Sulfurizing reduced the friction coefficient from about 0.4 to 0.2, while reducing wear by two-thirds. In comparison, nitriding led to only a modest improvement, and chromizing had adverse effects.

Plating It is theoretically possible to improve sliding characteristics by plating a thin layer of soft metal between two hard alloy surfaces. The soft metal is supposed to provide a low shear strength interface while preventing wear owing to direct contact between the hard alloys. In practice, however, results are difficult to predict. Chromium plating failed to improve the performance of AISI 304 stainless steel at 77 K [276]. Other

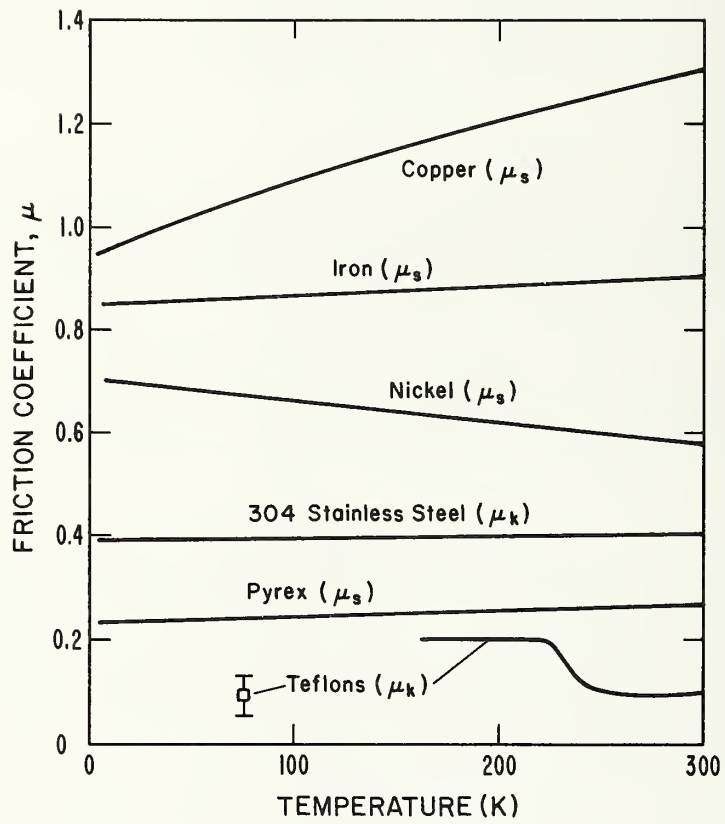


Figure 12.1. Temperature dependence of self-friction coefficients for selected materials.

tests show that lead, indium, or tin platings can be successful for AISI 440-C stainless steel in liquid hydrogen at 20 K for limited time periods [277]. For example, ion-plated lead coatings reduced wear significantly and yielded friction coefficients in the range 0.16 and 0.20, as opposed to 0.57 for unplated AISI 440-C. But Baber, Burton, Chang, Cuellar, Ku, and Russell [278] report high friction for lead-plated SAE 52100 tool steel at 77 K. Therefore, it appears that the effects of plating depend on the specific test conditions.

### 12.3.3. Laminar Solids

Certain solids having laminar (plate-like) crystal structures are easily sheared along their basal planes. This accounts for the high lubricity of graphite, boron nitride, and molybdenum disulfide ( $\text{MoS}_2$ ). Graphite and  $\text{MoS}_2$  are common dry lubricants at 295 K [279], but  $\text{MoS}_2$  is generally preferable at cryogenic temperatures.

Graphite is less expensive than other solid lubricants. But the lubricity of graphite in inert environments is poor, since the lubricity depends on the presence of water vapor or other absorbable gases. Thus graphite fails to lubricate in vacuum, and difficulties are encountered in some cryogenic environments where graphite has been shown to cause high friction and abrasive wear between sliding metals [280, 281].

Graphite is available in film form or as a component in mechanical carbons. In either case, low temperature tests have led to some erratic results. Baber, et al. [278] report an anomalous spike in the temperature dependence of the static friction coefficient for sprayed graphite coatings on copper, as shown in Fig. 12.2. Except for this anomaly, low static friction coefficients were observed in the cryogenic range ( $\mu_s \approx 0.1$ ). But the effects of prolonged sliding and wear were not investigated.

Molybdenum disulfide ( $\text{MoS}_2$ ), unlike graphite, is less sensitive to the environment. The reported friction coefficients for  $\text{MoS}_2$  in air at 295 K range between 0.04 and 0.1. At cryogenic temperatures, both high and low friction coefficients have been reported, depending on variables that are not completely understood.

$\text{MoS}_2$  can be added as a filler to plastics, or it can be used as a film coating for metals and plastics. When used as a film,  $\text{MoS}_2$  can be rubbed onto metal and alloy surfaces; it may also be sprayed, brushed, or adhesively bonded to metals or plastics. Bonded films endure longer, especially if the substrate is roughened or chemically etched prior to coating. On the other hand, the curing temperatures for certain commercial binders may be too high for some substrate materials.

Although superior static friction coefficients (0.1 to 0.2) are reported in Fig. 12.2 for  $\text{MoS}_2$  films on copper, continuous sliding at cryogenic temperatures may cause film deterioration. Wisander and Johnson [276] observed rapid film deterioration during wear tests of  $\text{MoS}_2$ -coated AISI 304 stainless steel at 77 K. The use of commercial  $\text{MoS}_2$  products in accordance with manufacturer's recommendations should minimize problems.

### 12.3.4. Polymers

In addition to moldability, machinability, and abrasion resistance, polymers show little tendency to adhere to metals. Thus nylon has replaced metals in some sliding applications such as gears, especially where freedom from corrosion is desired. A basic disadvantage of polymers is inability to dissipate frictional heat. This drawback is most evident for material in bulk form; it can be alleviated by utilizing filled polymers or film coatings, as discussed later.

In contrast to metals, the friction of polymers is more sensitive to load, temperature, and sliding speed; such variables may exert pronounced effects [272-290]. For example, Allan and Chapman [282] report sharp increases in the friction of Teflon at 300 K as load is reduced or as sliding velocity is increased; the low friction expected for Teflon is observed only at loads greater than 2.0 kg and at sliding velocities less than 25 mm/s.

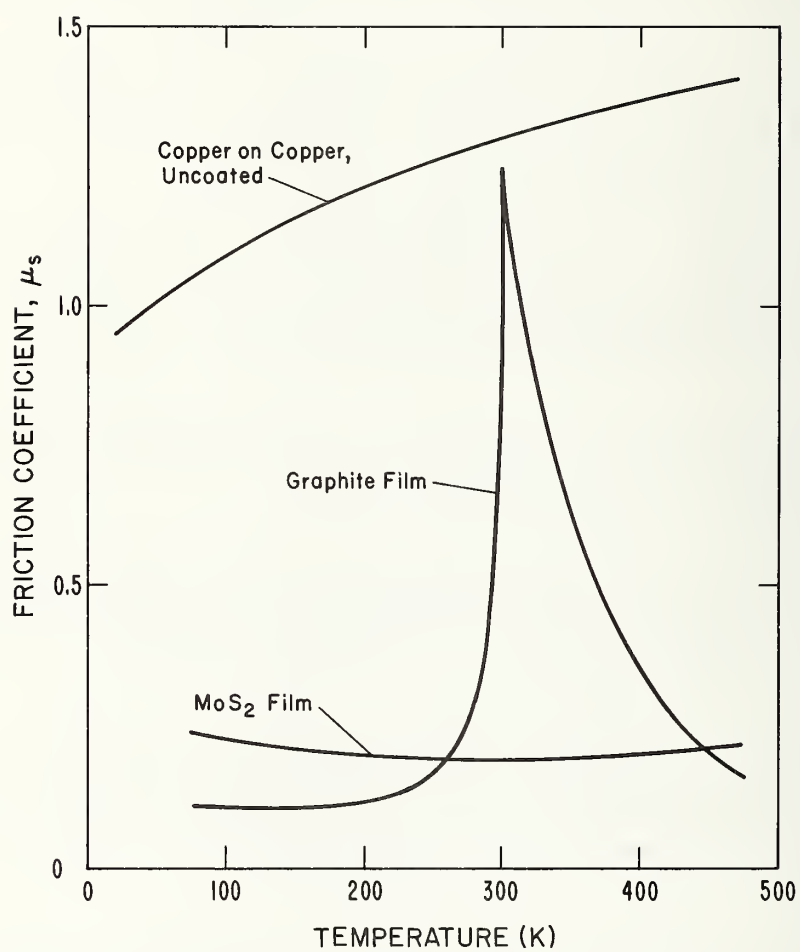


Figure 12.2. Effect of lubricating films on the friction of copper.



The room temperature static friction coefficients of polymers range from about 0.04 to 0.6. In most cases, the coefficients decrease at low temperatures. But Tabor [285] and White [290] agree that the static friction coefficient of PTFE shows a step-like increase from 0.1 at 253 K to 0.2 at 213 K. Outside this step region the coefficient is constant up to 350 K [282]. Friction coefficients in the range 0.06 to 0.13 have been reported for Teflon at 77 K, but no one has ever reported the complete cryogenic temperature dependence.

Kharitonova [286] reviews the cryogenic uses of Teflon (PTFE or FEP). Teflons have the lowest friction coefficients ( $\mu > 0.04$ ) of all known solids. Teflons are also exceptional in that they retain several percent ductility at 77 K, whereas other polymers are more brittle at this temperature. PTFE has a lower friction coefficient, is harder, and more wear resistant than FEP. If minimizing friction is not of overriding importance, other polymers having superior cold-flow resistance can be substituted for Teflons. Alternatives evaluated by Wisander and Johnson [289] include acetal copolymer, parapolyphenol, polyamide, polyimide, polycarbonate, polyoxymethylene, and polychlorotrifluoroethylene (PCTFE).

#### 12.3.5. Filled Polymers

Fillers can be blended with polymers prior to molding to achieve improved physical and mechanical properties. Diverse fillers have been used, including graphite, carbon,  $\text{MoS}_2$ , fibers, glass, pyroceram, ceramics, metals, and metal salts such as  $\text{CaF}_2$ . Typically, the filler amounts to about 25% of the total composition. Such a composite may offer significantly improved strength and thermal properties, while the antifriction properties of the bulk polymer are retained and problems associated with adhesive films (discussed below) are avoided. Filled polymers have been used as ball bearing separators [291].

A substantial data base exists to aid the selection of filler compositions for specific purposes. Friction and wear data for nine different polymers and seventeen filled compositions at 20 K are reported by Wisander and Johnson [289]. Studies devoted exclusively to filled Teflon compositions at temperatures down to 77 K were conducted by Timofeev, Drobinin, Grushevskii and Antropov [292], and by Wisander, Maley, and Johnson [275]. In the later study, 22 filled Teflon compositions were tested as rider specimens versus AISI 304 stainless steel. The friction coefficients varied between 0.06 and 0.13 at 77 K. Data are also presented for tests conducted in gaseous nitrogen at temperatures up to 344 K.

#### 12.3.6. Polymer Films and Coatings

Coatings of PTFE or PCTFE may be applied to metal components to combine the strength and thermal conductivity of alloys with the antifriction properties of polymers. Wisander and Johnson [293] report that 0.25 mm coatings of PTFE or PCTFE on AISI 304 stainless steel specimens give longer wear and lower friction compared with sulfurizing, chromizing, nitriding, chromium plating, and most solid-film lubricants. The success of a polymer coating depends on reliable application techniques, and to the long list of variables already cited as influencing friction and wear results, we must add two new variables — film thickness and bond integrity.

Coating failures are frequently caused by breakdown of the bond between the film and substrate. Wisander and Johnson's success was obtained by applying twenty 0.0127 mm thick Teflon layers and fusing at 644 K. In some cases this method may be impractical because of the high temperatures involved. An alternative, then, is adhesive bonding with organic resins, such as phenolics, epoxies, or silicones.

Regardless of bonding technique, spalling of the film from metal components is prone to occur since polymers expand and contract with temperature more than their metal substrates. For example, the average expansion coefficients between 77 and 300 K for Teflon and AISI 304 stainless steel are  $86 \times 10^{-6}/^\circ\text{K}$  and  $14.7 \times 10^{-6}/^\circ\text{K}$ , respectively. A practical solution was engineered by Wisander and Johnson [293], who formulated a 10% Teflon-40% epoxy-50% lithium-alumina-silicate film. The epoxy addition improved film adhesion, while the silicate reduced thermal contraction. The average coefficient of thermal expansion



for this film was  $28.8 \times 10^{-6}/^{\circ}\text{K}$ , which is close to values for metals and alloys. Tests verified that 0.127-mm-thick coatings of this film on AISI 304 stainless steel gave low friction ( $\mu = 0.02$  to  $0.07$ ), low rider wear, and outstanding film life. Another lubricating film for inert cryogenic environments is described by Sliney [294].

#### 12.3.7. Ceramics, Cermets, and Glass

The cold-welding of metals can also be avoided if at least one component of the friction couple is manufactured using a ceramic, cermet, or glass. These materials are corrosion resistant, wear resistant, and hard. They are also brittle at all temperatures, and will shatter if overloaded. Therefore, impact loading, high static loading, and high sliding velocities must be avoided.

Low temperature friction and wear data for these materials are scarce. Simon et al. [272] reported the static friction coefficient for fire-polished Pyrex glass to be constant at about 0.23 for temperatures between 77 and 300 K. Shynyrev and Drobinin [295] evaluated tungsten carbides at liquid nitrogen temperatures, reporting excellent wear resistance. Other materials of potential interest in this category include pyroceram, sapphire, and bonded oxides, such as  $\text{Al}_2\text{O}_3$ .

## 13. INSTRUMENTATION

The successful monitoring of cryogenic tests requires the appropriate selection and use of temperature, strain, and pressure sensors. In addition, proper technique must be observed in small voltage signal maintenance.

### 13.1. Temperature Sensors

Cryogenic thermometry reviews [296-300] include discussion of three main classes of temperature sensors for cryogenic service: (1) thermocouples, (2) resistance thermometers, and (3) filled systems. Representatives from each class are identified in Figure 13.1, and the performance characteristics of some common thermometers are compared in Table 13.1.

Copper-Constantan or Chromel-Constantan thermocouples appear to be the best general temperature sensor for cryogenic wind tunnel applications. Not the least consideration here is the need for small size and remote reading which thermocouples can satisfy. Platinum resistance thermometers and bondable temperature gages might also be useful, but other thermometers are of little or no value for NTF testing for reasons stated below.

#### 13.1.1. Thermocouples

Thermocouples are small, simple, convenient, fast in response time, readily available, well characterized, and low priced. The ancillary equipment required for use are low voltage measuring devices and reference junctions. Of the many possible types of thermocouple wire combinations, three standard types can be recommended for NTF testing: Types E, K, and T.

Type T (Copper versus Constantan). Type T is the traditional choice for service between 89 and 644 K. Owing to loss of sensitivity, Type T is not recommended for service below 76 K. Type T has high thermoelectric homogeneity, which means freedom from the adverse effects of material defects. Type T is the only thermocouple combination for which the limits of error are established below 273 K.

Type E (Chromel versus Constantan). This combination is recommended by ASTM for service between 23 and 1144 K. Sensitivity at the low temperature extreme is superior to Types T or K. Homogeneity is satisfactory, as is corrosion resistance in moist environments. Both wire elements have low thermal conductivity that reduces heat conduction to the specimen.

Type K (Chromel versus Alumel). This combination is recommended by ASTM for service between 23 and 1533 K. Sensitivity is nearly equivalent to type T, and only about half that of Type E. Both wire elements have low thermal conductivity and excellent corrosion resistance in moist environments.

The thermoelectric voltages, sensitivities, and other data for these thermocouples are given by several authors [301-305]. Procedures for installation, instrumentation, circuitry, and error analysis are treated in reviews [296-300].

#### 13.1.2. Resistance Thermometers

The platinum resistance thermometer (PRT) is the most accurate and reproducible thermometer available over a broad temperature range. Using the better commercial PRT's, wind tunnel temperatures can be measured routinely with high accuracy. Other resistance thermometers, however, must be dismissed from consideration: carbon and germanium resistance thermometers are restricted to service below 100 K, respectively, while copper and indium resistance thermometers simply lag the PRT in development at the present time.

The use of PRT's at low temperatures is discussed by Sinclair, Terbeek, and Malone [300]. The primary disadvantages are size and cost. Thermocouple wires are only 0.08 mm in diameter, but a PRT junction may be 1 x 8 x 10 mm. Ruggedness and reliability vary widely for commercial products, not necessarily correlating with price. Many configurations are commercially available, and custom designs can be ordered at additional cost. In general, however, all PRT's are classified as immersion probes or surface sensors.

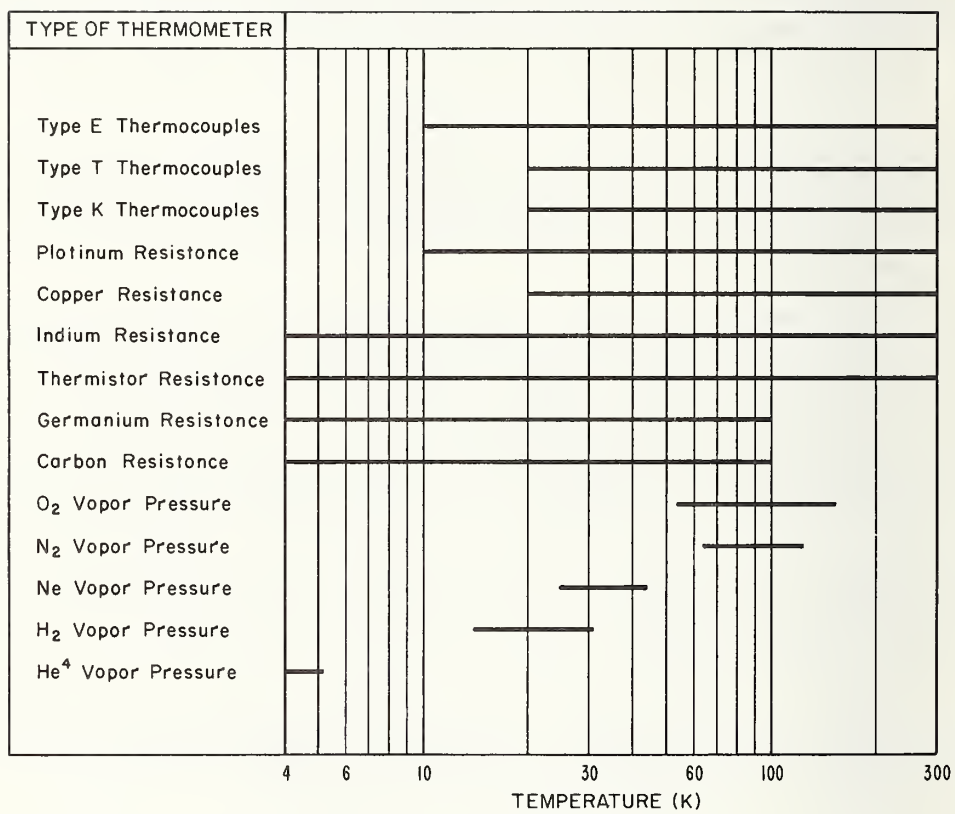


Figure 13.1. Thermometers and their useful temperature ranges.

Table 13.1. Operating characteristics of some selected thermometers.

Class	Type	Range K	Reproducibility K	Accuracy K	Response time s
Liquid-in-glass	Pentane	70-300	2	0.2-0.5	slow
Thermocouple	Copper-constantan	76-644	$10^{-1}$ - $10^{-2}$	0.1	~1
Resistance	Platinum	14-900	$10^{-3}$ - $10^{-4}$	$10^{-2}$ - $10^{-4}$	0.1 - 10
Vapor pressure	Oxygen	54-155	$10^{-2}$ - $10^{-3}$	0.02	0.1 - 100

Surface sensors are relatively small, typically 2.5 x 13 x 13 mm. Surface sensor geometries make good contact with various component shapes. They also have high custom design capability, and may be attached by clamping, soldering, welding, or cementing.

The variety of immersion probes is so great that product data bulletins must be consulted for specific descriptions. The typical immersion probe has a resistance somewhere between 100 and 1000  $\Omega$ . Repeatability at 273 K is typically certified conservatively at  $\pm 0.1^\circ\text{C}$ , but several thermal cycles to cryogenic temperatures are needed to establish repeatability. Time response is between 0.1 and 7 s.

Potentiometric voltage determination or direct resistance measurement can be used to measure PRT output. Appropriate bridge circuits are outlined by Harris [306], Stout [307] and Daneman and Mergner [308]. Both immersion probe and surface sensor PRT's are available in versions with built-in bridge circuits, which allow adjustments for improved interchangeability.

#### 13.1.3. Bondable Temperature Gages

Manufacturers of strain gages (described below) also produce bondable temperature gages for service between 76 and 513 K. These surface temperature sensors are constructed like strain gages, but the active elements are usually high purity nickel or nickel-iron foil grids. Accuracies equivalent to thermocouples can be achieved, but bonded temperature sensors are not reusable. Read-out methods, installation, stability, error analysis, and other information are described in product data bulletins.

#### 13.1.4. Thermistors

Thermistors (thermally sensitive resistors) are small, sensitive, and respond in less than 1 s. Unfortunately, few are designed for extreme cryogenic service, and each is usable only for narrow temperature ranges.

#### 13.1.5. Filled Systems

Pentane liquid-in-glass thermometers can be used at temperatures as low as 70 K. These thermometers require no calibration or instrumentation, but severe restrictions on use are imposed by the need for visual accessibility for direct reading. These thermometers may provide quick reference if positioned near wind tunnel viewing portals.

Vapor pressure devices function only for narrow temperature intervals and temperatures lower than 155 K, and are clearly inappropriate for wind tunnel service.

#### 13.1.6. Combined Thermometers

Thermometry capabilities can sometimes be increased by the combined use of thermocouples and resistance thermometers. Sinclair, Terbeek, and Malone [300] refer to several possibilities, including a case where the measurement of small temperature differences was improved using a thermocouple and a PRT.

### 13.2. Strain Gage Sensors

This section is exclusively devoted to electrical resistance strain gages which are directly bondable to machine components. Reference books on strain gages were authored by Perry and Lissner [309] and Aronson and Nelson [310], but these books do not discuss cryogenic applications. Current information is best obtained from the gage manufacturers.

Today's foil strain gages are produced in a wide variety of sizes, patterns, materials, and forms. One vendor boasts 40,000 variations, but not every kind is stocked. Most gages have a stated strain capability of 1% but this can usually be pushed higher, sometimes to 3%. Prices for photoetched films are about \$4 to \$10 each, depending on options. Options include protective encapsulation, solder dots, integrated terminals, preattached leads, high endurance leads, strippable backings, and self-temperature compensation.



A typical gage consists of an active alloy element and a dielectric backing, which provides easy handling as well as insulation. Backing materials include paper, bakelite, polyimides, epoxies, and reinforced plastic laminates. Active foil elements may be Constantan, Manganin, Karma, Nichrome, Chromel, platinum, nickel, Elinvar, or Armour D alloys. However, not every wire element and backing combination functions well at cryogenic temperatures.

#### 13.2.1. Gage Selection

Several gage element and backing combinations acceptable for various cryogenic operating conditions are identified by the manufacturers. "High performance" materials and construction are recommended for NTF applications since gage costs are minor compared to the overall research investment. The Karma alloy elements offer good apparent strain characteristics, stability, and fatigue life at temperatures down to 76 K. Armour D and Nichrome elements are also satisfactory at 76 K. Constantan is not generally applicable at cryogenic temperatures. Iso-elastic (Elinvar) elements are usable for dynamic testing between 76 and 500 K. Bondable terminals provide a stress relief point between the gage and lead wires, to avoid damaging the gage when lead wires are stressed. Satisfactory adhesives for bonding strain gages to all types of structural materials and test temperatures are available from the gage manufactures, as are cleaners, solders, applicators, measuring bridges, informative technical notes, and hardware kits.

#### 13.2.2. Gage Factors

The gage factor,  $G$ , indicates the sensitivity of the active element. Gage factors for the common cryogenic strain gage elements are approximately 2, but the value varies slightly with gage size, pattern, substrate, and temperature. In general, precise gage factors are furnished with commercial products, but it is possible to determine experimentally the temperature dependence of the gage factor using the relation:

$$G = \frac{\Delta R}{\epsilon R}$$

where  $R$  is the gage resistance and  $\Delta R$  is the change in resistance due to a strain,  $\epsilon$ .

#### 13.2.3. Apparent Strain

Unless precautions are taken, serious errors in strain measurements at cryogenic temperatures will arise from temperature effects on gage resistance, combined with thermal contraction of the materials involved. These errors give a false indication of strain, the magnitude of which can be much larger than the real strain.

Apparent strain must be accounted for to measure the true strain associated with mechanical stress. If the temperature is constant during the test, the problem is solved by bringing the gaged component to thermal equilibrium at zero load and then cancelling the apparent strain electrically. Or, if the apparent strain as a function of temperature for a gage mounted on a specific test material is published by the gage manufacturer, it can be subtracted from the total indicated strain to obtain the true strain. Alternative methods of eliminating apparent strain by using "dummy gages" are discussed in the technical notes published by gage manufacturers.

#### 13.2.4. Self-Temperature Compensation

Apparent strain may also be reduced using self-temperature compensating gages designed to minimize apparent strain when bonded to certain structural materials having matching thermal expansion and contraction coefficients. Generally, the best temperature compensation is achieved in the range 250 to 475 K. Detailed discussions of self-temperature compensation and the problem of apparent strain are to be found in technical notes published by the gage manufacturers.

### 13.3. Pressure Sensors

Pressure-sensing devices for cryogenic service are reviewed by Hayakawa [311] and by Arvidson and Brennan [312]. A number of commercial devices are suitable for NTF applications at pressures up to 900 kPa (9 atm). The simplest means of pressure measurement under static conditions is the bonded strain gage attached to a diaphragm. Pressure on the diaphragm stretches the gage, altering its electrical resistance. Although strain gages can be used for dynamic as well as static testing, piezoelectric devices offer superior frequency response in dynamic testing. These devices can be inserted by means of probes into the wind tunnel or into the model aircraft themselves.

### 13.4. Sensor-to-Specimen Contact

The object in bonding sensors to structural components is to achieve true physical contact, while minimizing the disturbance of the object being monitored.

For example, whenever a thermocouple is attached, a thermal barrier is created at the interface with the specimen, and heat must transfer between the specimen and sensor. The two major sources of heat are (1) heat conducted along the wires and (2) Joule heating ( $I^2R$ ) in the resistive thermoelectric elements. If the thermal barrier at the specimen interface is significant, heat flow across it will generate a temperature gradient. Consequently, the thermocouple may register a higher temperature than the true temperature of the specimen. As discussed in the following sections, thermal anchoring and contact agents can be used to alleviate the problem.

#### 13.4.1. Thermal Anchoring

Thermal anchoring (also called tempering) of wires to a heat sink whose temperature is near that of the specimen will allow heat to dissipate before it can affect measurements. Thermal anchoring is important in any cryogenic environment, but particularly in vacuum where there is no surrounding medium to absorb heat. Hust [313], Kopp and Slack [314] and Allen, Bradford, and Crabtree [315] discuss thermal anchoring in detail. The required anchoring length is a function of materials, wire size, insulation, temperature, environment, and the acceptable temperature mismatch of the wire and heat sink.

Thermal anchoring and other heat transfer calculations require knowledge of the thermal conductivity of wires, insulators, and contact agents (greases, adhesives, and varnishes). The thermal conductivities of some low temperature thermal contact agents for various temperature ranges are given by Denner [316], McTaggart and Slack [317], and Kreitman, Ashworth, and Rechowicz [318]. Denner and McTaggart and Slack also report thermal conductivity data for some electrical insulators and solders. Using discretion, engineering estimates of thermal conductivity for untested materials may be based on available data for similar materials.

#### 13.4.2. Thermal or Electrical Contact Agents

Contact agents are frequently used in cryogenics to attach heater wires, thermocouples, and heat sinks, or to improve electrical contacts. In the case of thermocouple attachment discussed above, electrical isolation must be maintained, preferably using a contact agent that is easily removed and resistant to thermal cracking. Greases, varnishes, and adhesives can be used, in conjunction with thermal anchoring.

Solders greatly reduce thermal barriers, but solders conduct electricity also. As electrical insulators, adhesives offer adequate thermal contacts with higher strength than varnishes or greases. Thermal conductivity and other useful data for adhesives and cements appear in the literature [217, 316, 319]. Thermal barriers can be further reduced by filling the adhesive with conductors such as silver or beryllium oxide. Kallin [320] reports a doubling of the thermal conductivity using a 65% epoxy and 35% silver powder mixture, as compared with the epoxy alone. This mixture was used to attach thermocouples to unweldable, brittle materials. The addition of silver did not decrease the adhesive bond strength significantly.

### 13.5. Signal Maintenance

Electrical signals from sensors must be maintained and processed without distortion. This is important since cryogenic instrumentation relies on low level signals. Wire insulation compatible with the cryogenic environment must be selected, and the introduction of spurious voltages must be avoided at wire junctions, electrical feed-throughs, ground current circuits, and switches.

#### 13.5.1. Wire Insulation

Wire insulation materials include polymers, varnishes, and glass fabric. These dielectrics retain their insulating properties at cryogenic temperatures. For example, polymer breakdown voltages are on the order of kilovolts. Of greater concern, therefore, is mechanical failure. Tensile, bending, or thermal stresses may crack wire insulation materials at cryogenic temperatures, causing electrical shorts.

Polymers are the most common form of wire insulation, and fluorocarbons are the recommended polymers. Based on simple mandrel winding tests at cryogenic temperatures, Teflon emerged superior in flexibility compared to polyethylene and polyvinylchlorides which are commonly used at room temperature [321]. Teflons and polychlorotrifluoroethylene are also preferred in aerospace cryogenic wiring applications by virtue of superior chemical inertness over a broad temperature range, as well as low dielectric loss. The FEP form of Teflon is readily available as spaghetti tubing or skived tape.

Regardless of the type of insulation selected, strain relief precautions should be taken during wiring. Note also that thin layers of insulation generally offer greater flexibility since higher strains are developed in thick layers subject to bending.

#### 13.5.2. Wire Connections

Wire connections should be limited to the minimum number practical. If a junction is made of dissimilar metals, it should not be located in a thermal gradient, or else spurious thermoelectric voltages will be generated. Note that solder junctions form a dissimilar metals situation, even when the two wires soldered are the same. Junctions can be made by soldering, spot welding, or simply twisting the leads together; all of these methods have unique applications and can be used successfully under proper conditions.

#### 13.5.3. Feed-Throughs

It is frequently necessary to route wires through walls where significant temperature and pressure changes occur in short distances. The two basic types of electrical feed-throughs used in cryogenic systems are the continuous wire and junction types as shown in Fig. 13.2. If a large temperature gradient exists across the feed-through, the continuous wire variety is preferable since it avoids a dissimilar metals situation. The specifications of materials, sizes, wire capacity, and pressure rating vary widely in commercial feed-throughs so that many practical applications can be satisfied.

Figure 13.2 also shows one of the many potting methods which may be used for "in-house" feed-throughs. The cup and reservoir design is simple and effective as an ambient temperature seal. If black sealing wax is used in the reservoir, the seal is easy to remake with new wires. Use of a continuous wire type eliminates the necessity of a junction, or at least allows the junction to be made at ambient temperature where large gradients do not exist.

#### 13.5.4. Shielding and Grounding

Care must be taken with low level voltages to obtain proper shielding or grounding. A single ground is preferred to avoid loop currents that can introduce spurious voltages in the signal path. Most modern low level dc amplifiers have excellent 60 Hz rejection; however, ac pickup may be sufficient to saturate the amplifier, resulting in a steady shift of output. The recommended procedures for low level dc circuits are:



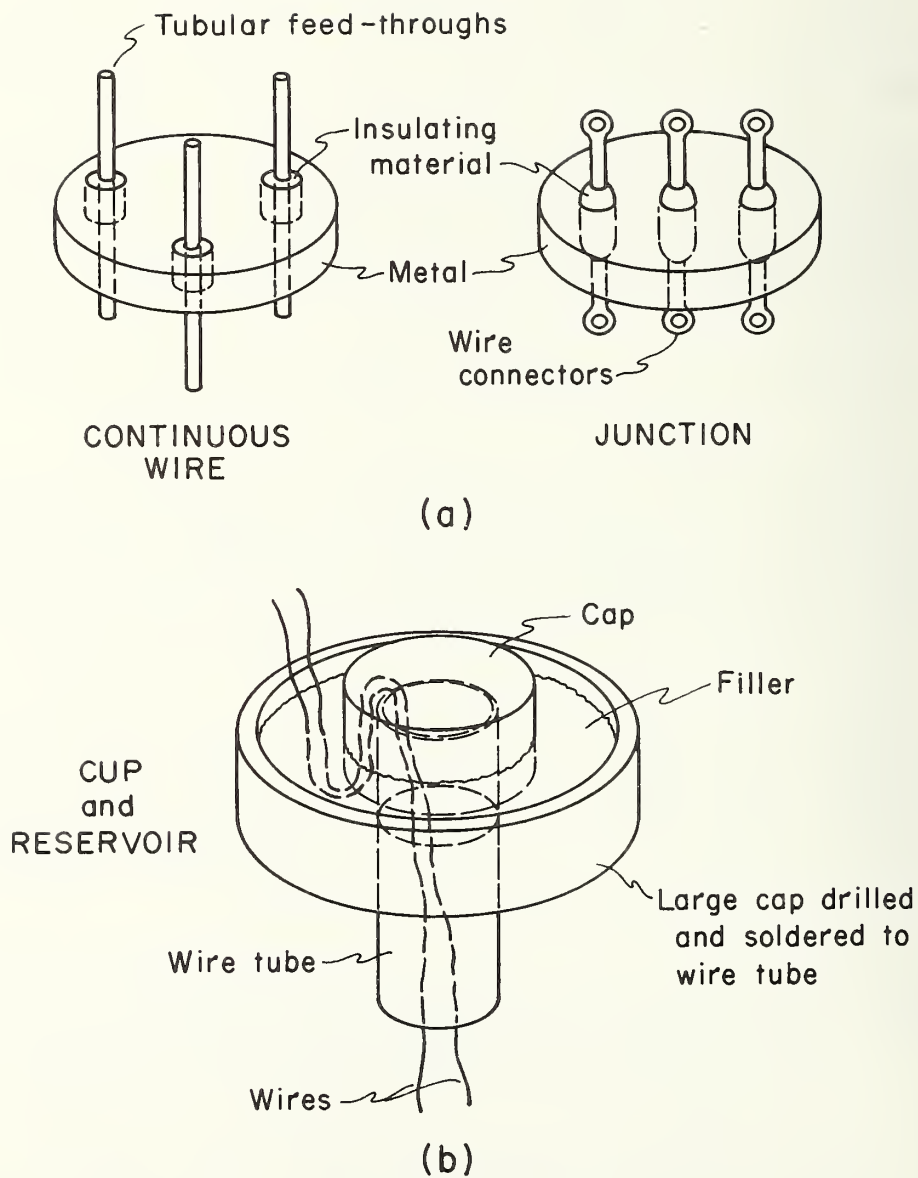


Figure 13.2. Various types of wire feed throughs used in cryogenic applications.

1) Operate either at floating potential or with one ground, preferably at the amplifier input.

2) Use extension cables that have a high rejection of electromagnetic noise.

Twisted leads decrease the flux pickup area, and commercially available weave patterns cause cancellation of pickup noise. The leads should be encased in a shield grounded at only one point. Since normal copper shielding does not adequately shield 60 Hz noise, cables having ferromagnetic shielding are preferable. Cables and wire junctions can be placed in iron conduits or boxes to provide additional electromagnetic shielding. Morrison [322] discusses shielding and grounding in greater detail.

#### 13.5.5. Switching

Switches can be operated at ambient temperatures where problems of spurious voltage generation are not severe, but they must be of sufficient quality to prevent degradation of the signal-to-noise ratio. For manual operation, low-resistance rotary switches in metal cabinets are satisfactory. For automatic operations, enclosed gold- or silver-plated switches are usually satisfactory; they typically have about 1 mV transient noise and therefore introduce excessive noise if switched faster than about once a second. All switches should be thermally isolated since thermal voltages may be generated at junctions where wires are attached.



## 14. SOURCES OF CRYOGENIC DATA

In a survey of this kind, it is not possible to describe all materials and parameters that may be of potential interest for specific applications. New materials and measurements are appearing at a high rate. To keep abreast of developments, several data sources dealing with cryogenics are described below. Cryogenic handbooks [8-17] have been recommended earlier in the text. An additional aid to data retrieval from sources possibly containing some cryogenic data is the survey by Westbrook and Desai [323] which attempts to assess the content of the most prominent handbooks, technical compilations, information centers, technical societies, and trade associations.

### 14.1. Technical Journals

Cryogenics (Plenum) is an international journal covering all aspects of low temperature research. Cryogenic Technology (Cryogenic Society of America) and Cryogenics and Industrial Gases (Business Communications, Inc.) are primarily of technological interest. Cryogenic Information Report (Technical Economics Associates) surveys current activities and developments in all fields of cryogenics.

### 14.2. Monographs

The International Cryogenics Monographs Series (Plenum) at present consists of eleven volumes. Of these, Wigley's [18] Mechanical Properties of Materials at Low Temperatures is of greatest relevance to model designers. The National Bureau of Standards also publishes monographs devoted to cryogenics. A complete listing of published NBS titles is available from Frizen and Mendenhall [324].

### 14.3. Conference Proceedings

Advances in Cryogenic Engineering (Plenum) contains the proceedings of both the International Cryogenic Materials and Cryogenic Engineering Conferences, which are held every two years. There are currently 24 volumes. Volume 20 contains an index to all previous volumes.

Other sources include six volumes of proceedings from the International Cryogenic Engineering Conferences (IPC Science and Technology Press) and seven Special Technical Publications (STP's) from the American Society for Testing and Materials:

- ASTM STP 158 (1954). Symposium on the Effect of Temperature on the Brittle Behavior of Metals with Particular Reference to Low Temperatures.
- ASTM STP 287 (1961). Low Temperature Properties of High Strength Aircraft and Missile Materials.
- ASTM STP 302 (1962). Evaluation of Metallic Materials in Design for Low Temperature Service.
- ASTM STP 387 (1966). Behavior of Materials at Cryogenic Temperatures.
- ASTM STP 496 (1971). Fracture Toughness Testing at Cryogenic Temperature
- ASTM STP 556 (1974). Fatigue and Fracture Toughness - Cryogenic Temperatures.
- ASTM STP 579 (1975). Properties of Materials for LNG Tankage.

### 14.4. Computerized Data Banks

Bibliographies on specific subjects may be purchased from organizations maintaining automated data bases and search systems. The following information on data bases is quoted from Fickett [325].

1. NTIS - covers abstracts from U.S. government sponsored research by 240 agencies and their contractors. Dates from 1964. Updated biweekly. Contact: U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161. This service also publishes a separate section of its Weekly Government Abstracts under the title "Materials Sciences". Cryogenic applications are not separately indexed, although they appear occasionally. Available by subscription.

2. SSIE - summaries of research in progress and recently completed (U.S. and foreign). Dates from 1974. Updated monthly. Contact: Smithsonian Science Information Exchange, Room 300, 1730 M Street, Washington, D.C. 20036.

3. COMPENDEX-- Engineering Index data base. Abstracts of published work from 3500 journals, reports and books. Dates from 1970. Updated monthly. Contact: Engineering Index, Inc., United Engineering Centre, 345 East 47 Street, New York, NY 10017.

4. METADEX - covers abstracts of the international metallurgical literature. Dates from 1966. Updated monthly. Contact: Metals Information. American Society for Metals, Metals Park, OH 44073.

5. NASA maintains a computerized data base from its Scientific and Technical Aerospace Reports (STAR) abstract journal, but the situation regarding general access is not clear. The published version of STAR is generally available. Contact: NASA Scientific and Technical Information Facility, P.O. Box 8757, B.N.I. Airport, Maryland, 21240.

In addition to the above, the Cryogenic Data Center, Thermophysical Properties Division, NBS, Boulder, Colorado, 80303, also prepares tailored bibliographies based on an automated indexing system. The minimum service charge is \$35.00. The Cryogenic Data Center is a prime source of cryogenic information, maintaining a library and world literature file with over 100,000 entries open to the visiting public. Out of print, or hard-to-locate reports are included.

#### 14.5. Abstracting Services

The Current Awareness Service (Cryogenic Data Center, NBS, Boulder, Colorado, 80303) is a biweekly publication listing an average of 200 cryogenic articles per issue. Based on a scan of 1,000 periodicals, the content is divided into sections on Low Temperature Physics and Chemistry, Cryogenic Engineering, and Energy. The annual cost is \$25.00.

The Review of the Low Temperature Properties of Metals (Metals Properties Council and Metals and Ceramics Information Center, Battelle-Columbus, Columbus, Ohio, 43201) also provides a convenient summary of low temperature research studies appearing in the open literature and in private reports.

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